RESEARCH AND TECHNOLOGIC WORK ON COAL AND RELATED INVESTIGATIONS, 1958

By Staff, Division of Bituminous Coal

* * * * * * * * * * * * information circular 7970

UNITED STATES DEPARTMENT OF THE INTERIOR
Fred A. Seaton, Secretary

BUREAU OF MINES
Marling J. Ankeny, Director

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RESEARCH AND TECHNOLOGIC WORK ON COAL AND RELATED INVESTIGATIONS, 1958[1]

by

Staff, Division of Bituminous Coal

INTRODUCTION AND SUMMARY

This report is the 23d in a series summarizing research and technologic work by the Federal Bureau of Mines on coal and related investigations; it is the fifth report to be issued on a calendar-year basis.[2] Publications by Bureau personnel in 1958 are cited;[3] in addition work in progress during the year is briefly described.

To conserve our reserves of coal the Bureau of Mines conducts a program concerned with the development of fundamental information on coal characteristics and behavior, and the improvement of technology related to its production and utilization. The ability to anticipate and meet future demands for coal depends on a number of factors: (1) Knowledge of reserves from a standpoint of both quantity and quality; (2) the development of improved mining methods, health and safety measures, and preparation techniques that will insure its most efficient exploitation; and (3) knowledge of the controlling factors in efficient use of coal in combustion, carbonization, gasification, direct and indirect hydrogenation, and other new processes. The Bureau is concerned with each of these areas.[4],[5]

1/ Work on manuscript completed October 1958.

2/ Bureau of Mines Staff Research and Technologic Work on Coal and Related Investigations, 1957: Bureau of Mines Inf. Circ. 7905, 1959, 85 pp.

3/ Carman, E. P., and Bayes, F. S., Bibliography of Bureau of Mines Investigations of Coal and Its Products, 1950 to 1955; Bureau of Mines Inf. Circ. 7825, April 1958, 135 pp.

4/ Scollon, T. R., Are We Really Running Out of Fossil Fuels? Power Industry, vol. 74, No. 6, June 5, 1958, pp. 12-13.

5/ Perry, Harry, Coal's Future in the Energy Market, Mech. annual, December 1958, pp. 46-60.

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The Bureau's work was performed at the following Stations:

Station	Location	Main coal activities
Headquarters	Washington, D. C.	Programing and budgeting; foreign activities; Government fuel-engineering service.
Anthracite Experiment Station	Schuylkill Haven, Pa.	Anthracite investigations.
Region I		
Alaska Mining Exp. Sta.	Juneau, Alaska	Exploration.
Anchorage Exp. Sta.	Anchorage, Alaska	Sampling and analysis.
Northwest Exp. Sta.	Seattle, Wash.	Preparation.
Region III		
Denver Exp. Sta.	Denver, Colo.	Western coals investigations.
Lignite Exp. Sta.	Grand Forks, N. Dak.	Lignite investigations.
Region V		
Central Exp. Sta.	Pittsburgh and Bruceton, Pa.	Health and safety investigations (District B); mining, preparation, combustion, carbonization, hydrogenation, petrography and constitution, sampling and analysis of bituminous coal.
Appalachian Exp. Sta.	Morgantown, W. Va.	Gasification, low-temperature tar.
Eastern Exp. Sta.	College Park, Md.	Boiler-water research and service; stream and air pollution.
Southern Exp. Sta.	Tuscaloosa, Ala.	Preparation and carbonization.
Gorgas Exp. Sta.	Gorgas, Ala.	Underground gasification.

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Among the services furnished to Government agencies are sampling and analysis of coal from current production, the results of which are used in awarding contracts by Federal agencies. During the year 916 tipple samples were collected at 344 mines in 16 states, and a report of these analyses was published, the 8th in this series. Assistance was also given in establishing standardized sampling procedures for coal delivered to Government installations. The analytical work involved in the evaluation of samples resulted in approximately 258,000 chemical and physical tests on about 48,000 samples.

Since World War II, international trade in coal has increased greatly and emphasized the need for international classification of coal. The Bureau has cooperated with the Coal Committee of the Economic Commission for Europe in applying to American coals the system adopted in 1956 for classification of coals higher in rank than lignite, and in developing a system for the international classification of brown coals and lignite. In conjunction with this work techniques used for coal analysis abroad were assessed, and their application to American coals was evaluated.

Revision of standard methods and development of new techniques for determination of ash, mineral matter, and other characteristics of coal and derived products are being studied.

Methods for studying the structure and chemistry of coal have progressed in recent years. The application of modern analytical tools, including evaluation of the optical properties of coal by indexes of refraction and adsorption, and infrared, visible, ultraviolet, and X-ray spectra have proved to be useful tools. The physical structure of coals has been studied by means of adsorption to determine surface areas and pore diameters. Coals have been separated into relatively pure petrographic components, and the differences in physical and chemical properties of the petrographic constituents are being investigated. Two new methods for hydrogenating coal at low temperatures were studied further, and the results indicate that the study of the reaction products may help to elucidate the chemical structures present in coal. The microbial degradation of coal was studied further as another means for obtaining products from coal that have not been altered by the application of heat. Studies of the bacteriostatic and fungistatic materials found in coal have indicated that preparations obtained by solvent extraction contain antibiotic materials.

Fundamental and applied research was conducted to determine and take full advantage of the unique properties of anthracite in developing large-tonnage uses. The fundamental work was concerned chiefly with extending present information on the basic physical and chemical characteristics of anthracite. Two projects were begun, one to determine the chemical structure of anthracite by oxidation with nitric acid and the other to determine the effects of neutron radiation on anthracite.

In response to widespread interest in the effects of high-intensity nuclear radiation on coal, a program was initiated to study the effects of irradiation. Samples were prepared for exposure, and plans were made to analyze the products.

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The Bureau has participated in a program to develop Alaskan coal reserves. In the last fiscal year core drilling in the Moose Creek area of the Matanuska coalfield was continued and a reconnaissance of the Beluga River coalfield was made.

A program was initiated to investigate a new mining method. Equipment for hydraulic mining of coal was assembled at the site selected for preliminary tests. A number of other programs designed to improve the efficiency of mining operations and to reduce mining costs was continued. In anthracite mining, efforts were made to overcome the difficulties of mining pitching beds and to adapt high-productivity coal-mining machinery to these conditions, to develop low-cost long-wall systems, and to modify current mining systems to permit increased mechanization. A program on hydraulic hoisting of anthracite was initiated. In bituminous coal mining, studies were continued of methods and equipment for underground development and face haulage; the comparison of cost of timber and roof bolt supports; and examination of mine records to determine the amount of coal recovered as a function of the mining method.

Bureau representatives gave technical assistance in cooperative coal projects with the governments of Afghanistan, Colombia, India, and Indonesia in 1958, with funds made available by the International Cooperative Administration.

Health and safety activities were continued, and the collection and publication of statistics on accidents and fatalities, as well as other related studies, have pinpointed areas where further research in mining methods is needed.

Research into problems of roof support continued in 1958. An investigation of bumps or coal outbursts in coal mines was started during the year.

Safety standards to prevent hazardous conditions associated with auger mining were recommended. Research was continued on mine lighting and mining equipment related to increasing safety.

As part of the Bureau's coal mine inspection under the provisions of the Federal Coal Mine Safety Act, approximately 18,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines. Other studies of ventilation problems confronting the coal mining industry were continued.

A program was initiated in the year to develop a continuous methane monitoring system. Hot-wire oxidation of methane and modifications of this method are being studied. A tentative list of specifications for methane monitoring systems was prepared as a guide for manufacturers.

The program of safety training and education was also continued.

Mined coal is often stored before use, with the period of storage varying greatly. Storage of low-rank coal can create a number of problems, particularly in regard to the tendency of these coals to heat spontaneously. The

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effect of storage on lignite was observed on the 2 million tons successfully stockpiled since 1953 at Garrison Dam, N. Dak. Pelletizing techniques were also investigated as a method for producing a stable storable fuel from small-particle lignite of limited marketability obtained during the preparation of commercial sizes. The effect of process variables on the properties of the pelletized material was investigated.

Because of severe winter conditions in the major lignite-producing area and the inherently high moisture content of lignite, freezeproofing is an important problem. Techniques for the prevention of lignite freezing have been evaluated; the commercial spray oil technique currently used and the admizing of air-dried lignite to the as-mined product appear to be the most effective means of reducing agglomeration.

In its natural state, lignite is a tough and resilient material that is resistant to fracture. The size reduction of lignite is therefore being studied.

Mixing and segregation are of great importance in all phases of coal preparation and utilization. A comprehensive investigation of this problem is being conducted, with small-scale laboratory studies of model systems. A mathematical criterion was developed during the year that provided a convenient measure of the degree of mixing of multi-component systems.

Since the advent of large-scale mechanized mining, coal preparation has encountered new problems. Cleaning and dewatering of fine coal are the most difficult aspects of coal preparation and these receive intensive attention. Cleaning of coarse sizes of coal by dense mediums is also being studied. Surveys of the preparation of coking coals by counties and a study of reduction of ash in anthracite were continued.

Preliminary tests with the feldspar jig indicated effective cleaning down to 48 mesh. The dense-medium cyclone is another fine-coal cleaning apparatus being studied. A pilot plant was installed to treat unsized coal to eliminate separate flotation cleaning of the extreme sizes. Evaluation of the Sieve Bend (the DSM screen) was continued, and high capacity and sizing efficiency were obtained in laboratory tests with a commercial size unit. The removal of water from fine coal by the use of a vacuum filter and radiant heat was studied in preliminary tests which indicated that the cost per ton of water removed by this technique is about one-half the cost of thermal drying.

The survey of washability characteristics of coals on a county basis, begun in 1948, was continued. Reports for 2 Virginia counties were published in 1958, bringing the total completed to 23 counties.

Generation of heat and power by combustion has been the primary use of coal for centuries. However, problems involved in its most efficient utilization remain unsolved. One of these problems is heat transfer in furnaces and its dependence on flame temperature and ash deposits on the external surfaces of boiler tubes. This problem is being studied and includes analysis of data from commercial installations, a study of the nature of the deposits, and an

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investigation of the basic factors involved in heat transfer. The fluid mechanics involved in furnace operation are not yet fully understood, and this subject is also under investigation. The combustion of anthracite and lignite was also studied. Standard American Society of Mechanical Engineers tests conducted on four different small industrial anthracite stokers showed efficiencies of over 80 percent under continuous stoker operation.

The Bureau provides fuel engineering services to other Federal agencies to increase efficiency in the use of coal. Performance and efficiency tests were made on steam generating and auxiliary equipment. In conjunction with boiler-feedwater service to Federal heating and power plants, recommendations were made on boiler water treatment to minimize difficulties arising from condensate corrosion. The Bureau-developed condensate-corrosion tester is widely used to determine the need for corrective measures and to evaluate the effectiveness of filming treatment. The evaluation of filming with octadecylamine was continued.

Combustion produces materials that contribute to air pollution, and methods of reducing these pollutants are being studied. Incinerators for the disposal of normal and radioactive waste have been developed and are being tested. Methods for removing sulfur dioxide from flue gases were evaluated. The development of catalysts for oxidation of automotive exhaust gases was investigated, and plans were made to test the most promising preparations using exhaust from a single-cylinder engine.

Work on coal carbonization includes studies of the mechanism of thermal decomposition, process variables, and assays, by both low- and high-temperature methods. A fluid bed unit is used to produce primary tars, which are then passed through a cracking unit maintained at controlled higher temperatures to study conditions that determine the composition of tar obtained in conventional coke ovens. The dependence of coke structure on the petrographic composition of the coal used is also being studied. Studies of the effects of inerts in coal, of pretreatment of coal before its softening temperature, and of the properties of the resulting coke were continued. Tests to determine the effect of preheating on the carbonizing properties of the coal indicated that weaker coking coals were improved appreciably as a result of preheating, whereas the more strongly coking coals and blends showed little effect.

A comparison was made of the physical properties of commercial cokes and experimental cokes produced by laboratory methods. To evaluate the interrelationships among coke quality, oven width, and coking rate, physical tests were made on cokes produced at different rates and at 3 oven widths in tests at two or more flue temperatures. The results indicate that the changes in flue temperature and oven width had little or no effect on the properties of cokes produced at the same constant coking rate.

The cooperative research program on improving the quality of coke made from western high-volatile bituminous coal was concluded in 1958. The study included a total of 52 high-temperature carbonization tests in the 500-pound, 14-inch slot-type coke oven.

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The BM-AGA (Bureau of Mines - American Gas Association) carbonization assay developed many years ago is used for assessing the value of coking coal reserves by surveying the carbonizing properties of coals from the principal American coalbeds. Seventeen Pensylvania and 13 West Virginia coals were tested. Two additional reports on coals from counties in the Appalachian region were published in 1958.

Anthracite is currently used by the iron and steel industry in blends with bituminous coal for making metallurgical coke, in sintering and pelletizing iron ore, and as a carbon source. Studies are in progress to extend its use by the metallurgical industries, especially as a blast-furnace and cupola fuel. Calcining anthracite to increase its resistance to thermal shock is one of the main subjects being investigated. Calcined anthracite was tested in three foundries as part of this activity.

The production of anthracite metallurgical briquets, followed by thermal stabilization was studied further. A pilot briquetting plant with a capacity of approximately 1-1/2 tons per hour and a coal drier were built during the year. Tests were begun to develop information on the economics of the process and to prepare enough briquets for trial in commercial metallurgical equipment.

Carbonization of coal at temperatures lower than those used in conventional coking practice produces a char that is more reactive than blast furnace coke, and also increased yields of tar which may prove to contain useful chemicals. The small-scale carbonization assay reported in 1953 is used as a bench-scale test and research tool. Carbonization studies of western coals were continued as were further studies of drying and carbonizing fine coal and Texas lignite in an entrained bed. Preparations were made to study pressure gasification of lignite; carbonization of lignite at elevated pressures was studied.

The Bureau completed its program of technical assistance to the Indian Government in connection with utilization of lignite from Neyveli, South Arcot. The pilot plant was built in 1958, and operations and tests were begun.

Tar obtained under the relatively mild conditions of low-temperature carbonization reflects the composition of the original coal to a much greater extent than tar from conventional high-temperature coking. Separation and characterization of the constituents of low-temperature tars from lignite and bituminous coals are part of the Bureau's program, since the economic success of low-temperature carbonization depends on producing tar that has a value greater than its fuel value. Detailed studies of the phenols, bases, and neutral oils in the tars were continued. Upgrading of certain fractions is being studied as a possible means for producing materials of commercial interest. New techniques designed to facilitate this work have been developed and used.

Gasification of coal will increase in importance as the reserves of natural gas and petroleum decrease. Hydrogen is required for direct hydrogenation of coal to liquid products, and mixtures of hydrogen and carbon monoxide are needed for indirect hydrogenation and for producing high-B.t.u. gas.

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Studies are in progress on the rates and mechanisms of the gasification reactions and on problems associated with the operation of various types of gasifiers. Production of hydrogen by the steam-iron process, in which iron is alternately reduced by producer gas and oxidized by steam, is being investigated as an alternate to the coal-steam-oxygen reaction for converting coal to gas.

Because of its inherently high reactivity, lignite appears expecially suitable for gasification. A fixed-bed pressure gasification process is therefore being developed for the gasification of lump lignite with oxygen and steam. A gasifier has been designed to operate under slagging conditions, with provisions for conversion to dry-ash-removal, and the pilot-plant was 95 percent completed.

Gasification of pulverized coal in an entrained state has been studied for several years, and attempts have been made to improve operations. One of the major problems, feeding pulverized coal to a pressurized system, has been investigated by several approaches.

Another study is related to the possibility of applying nuclear energy to supply the heat required for coal gasification; a simulated nuclear reactor, heated electrically, is being used for preliminary tests of materials of construction and component life. Tests with a helium loop used for heat transfer were made at atmospheric pressure. An associated problem, the purification of helium, was studied; a tester is being devised to determine trace impurities in helium, and methods are being developed to reduce these impurities.

Data from anthracite gasification tests in Germany were analyzed and used as the basis of an engineering study on the production of high-B.t.u. gas, synthesis gas, and hydrogen from anthracite.

Underground gasification of coal was explored further. Preliminary cost estimates, however, indicate that energy so recovered is not economically competitive (with present technology and under present circumstances) with that derived from conventionally mined and burned coal.

For direct use or for synthesis purposes, gas from coal must be purified to remove gaseous and solid constituents that may be detrimental to the desired objective. Methods of purification are being investigated, and a technique developed by the Bureau for scrubbing gas by hot solutions of potassium carbonate has found industrial application.

The two principal methods for converting coal to fluid fuels are direct reaction of coal with hydrogen at elevated temperatures and pressures; and gasification of coal to mixtures of carbon monoxide and hydrogen, followed by catalytic transformation of these gases to the desired products. Both methods are being studied by the Bureau in fundamental laboratory work, bench scale, and pilot plant operations. In direct coal hydrogenation, the autoclave is used to evaluate catalysts and process variables. Pilot plant work has been concerned with the conditions for maximum production of distillate fuels at low pressure, 1,500 to 3,500 p.s.i.; and hydrogenating coal at high tempera-

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tures, 800°C., with short residence times. Development of methods for characterizing the products is an integral part of this work. Mass spectrometric analysis with low ionizing voltages was applied to the characterization of coal hydrogenation products.

In the Fischer-Tropsch synthesis of liquid fuels from carbon monoxide and hydrogen, major problems are encountered in preparing catalysts that have a satisfactory life, are resistant to poisons while yielding products of a desired composition, and have satisfactory activity. Studies on these problems were continued. Knowledge of the chemistry of metal carbonyls has contributed significantly to an understanding of the reactions involved. The Fischer-Tropsch synthesis releases large amounts of heat, and this fact introduces major problems in the design of reactors to maintain temperature control and to use the evolved heat effectively. One of the techniques developed and explored on a pilot plant scale involves a gas recycle process that offers considerable promise.

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HG & M Coal Co.
Hanna Coal Co.
Harmar Coal Co.
Harmon Creek Coal Corp.
Haynes Stellite Co.
Hudson Coal Co.
International Nickel Co.
Jeddo Highland Coal Co.
Jimmies Coal Co.
Jones & Laughlin Steel Corp.
Kaiser Steel Corp.

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Kennametal, Inc.
Knife River Coal Mining Co.
Koppers Co., Inc.
Lehigh Coal & Navigation Co.
Lehigh Valley Coal Sales Co.
Lenzi Coal Co.
Mallinckrodt Chemical Works
Mathies Coal Co.
McDanel Refractory Porcelain Co.
Minnesota Mining and Manufacturing Co.
Minnkota Power Cooperative, Inc.
Natali Coal Co.
National Carbon Co., Div. of Union Carbide Corp.
National Mines Corp.
Northern States Power Co.
Norton Co.
The Pacific Coast Co.
Panrell Coal Co.
Partazana Coal & Construction Co.
Pittsburgh Coal Co., Div. of Consolidation Coal Co.
Rand Mining Co.
Raven Run Coal Co.
Reading Anthracite Co.
Red Diamond Mine, Zeigler Construction Co.
Rochester & Pittsburgh Coal Co.
Rosedale Coal Co.
Rosedale Mine, Bethlehem Mines Corp.
Russell Manufacturing Co.
Sanderson and Porter
Saskatchewan Research Council
Shaffer Coal Co.
Stacy Coal Co.
State Line Coal Co.
Susquehanna Collieries Div., M. A. Hanna Co.
Texas Power and Light Co.
Textile Machine Works Co.
Trent Tube Co.
Truax-Traer Coal Sales Co.
Tube Methods, Inc.
Union Carbide Corp.
U.S. Army Corps of Engineers
U.S. Atomic Energy Commission
U.S. International Cooperation Administration
U.S. Pipe and Foundry Co.
U.S. Public Health Service
U.S. Steel Corp.
University of Alabama
University of North Dakota
University of Washington
Valley Camp Coal Co.
Weirton Coal Co.
West Virginia University
S. S. White Dental Manufacturing Co.
Williams River Coal Co.
Zirconium Corp. of America

ORIGIN AND PROPERTIES OF COAL

Inspection, Sampling, and Analysis

An inspection, sampling, and analysis service is maintained by the Bureau for all Federal agencies to facilitate their purchase of coal on specifications and bids. The analyses of tipple and delivered samples accumulated in the course of this work are made available to the public and are widely used in the purchase of industrial coal. The eighth of a series of publications issued annually covering analyses of tipple and delivered samples of coal collected throughout the United States, including Alaska, was released.6/ The analyses are arranged alphabetically with respect to states, counties, towns and mines and are listed as follows: Proximate analysis; ultimate analysis; calorific value; ash-softening temperature; free-swelling index; and Hardgrove grindability

6/ Aresco, S. J., Haller, C. P., and Abernethy, R. F., Analyses of Tipple and Delivered Samples of Coal (Collected During the Fiscal Year 1957): Bureau of Mines Rept. of Investigations 5401, 1958, 59 pp.

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index. The tipple samples were collected by engineers of the Bureau of Mines at the mine tipple or breaker as the coal was loaded into railroad cars and trucks. The delivered samples were collected systematically throughout all deliveries by representatives of the Government agencies under directions supplied by the Bureau of Mines.

In addition to the regular series of Reports of Investigations in which the analyses of tipple samples and coals delivered to government agencies are published, a bulletin on the analyses of Washington coals was issued.7/ Information on the Washington coal fields, description of mines, and statistical data on preparation, production, distribution, and use are included.

During the year, coal was produced equal to or in excess of demand and Federal agencies had no difficulty in obtaining bids covering their requirements.

Coal requirements of Federal agencies for 1958, excluding the Atomic Energy Commission and Tennessee Valley Authority, were estimated at approximately 5 million tons. All agencies, with the exception of the Post Office Department purchased their coal on a guaranteed-analysis basis, including penalties for delivery of substandard coal. Requests from Federal agencies for analyses for use in evaluating coal bids continued about the same rate as in the previous year. The Navy Department inaugurated a new policy of contracting for coal. Instead of concentrating purchases in the winter and spring months for deliveries in fiscal year 1959, purchases were spread out throughout the calendar year 1958, with contracts running through fiscal year 1960. Recommendations were made for award of contracts by some Agencies.

Proximate or ultimate, sulfur, and calorific analyses were reported on 8,754 samples, a decrease of 19.8 percent from the calendar year 1957; ash-softening temperatures were reported on 2,166 samples; free-swelling index on 369 samples; and Hardgrove grindability on 254 sampels in connection with Government coal purchases.

Analyses were reported on 916 tipple samples collected at 344 mines in 16 states, and on 144 samples of export coal, representing approximately 226,000 gross tons, being shipped overseas by the General Services Administration.

Other samples were analyzed or tested in connection with Bureau of Mines investigations of safety in mining, and of preparation and utilization of coal. These samples included 70 coals that were tested for estimation of coking properties by means of one or more of the following bench-scale tests: Agglutinating value test, Gieseler plastometer test, Audibert-Arnu dilatometer test, and the Fischer-Schrader low-temperature carbonization assay. In connection with a study of the formation of ash deposits on boiler tubes, chemical analyses were made of 65 samples of fly ash and related materials. Seven carbonization samples and fourteen preparation samples were collected at mines in Pennsylvania and West Virginia.

7/ Daniels, Joseph, Yancey, H. F., Geer, M. R., Abernethy, R. F., Aresco, S. J., and Hartner, F. E., Analyses of Washington Coals - Supplement to Technical Papers 491 and 618: Bureau of Mines Bull. 572, 1958, 92 pp.

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Under the working agreement with the Atomic Energy Commission, 417 samples of delivered coal were collected and prepared at the Savannah River Operations Plant, Aiken, S. C. At the request of the Marine Corps, stockpile samples at the Marine Corps Training Center, Parris Island, were collected and prepared.

Personnel of the Northwest and Anthracite Experiment Stations were instructed in proper coal sampling procedures and in collecting export samples. At the request of the Air Force, a coal sampling school was held at Mitchell Air Force Base for personnel of the Continental Air Command. The success of this school stimulated the Air Force to schedule four schools for 1959.

Meetings were held with representatives of the various Army commands at Washington, D. C., to review coal sampling techniques and coal requirements for fiscal year 1959.

The Bureau of Mines Experiment Station at Anchorage, Alaska, provides analytical, sampling, and inspection service to facilitate the procurement and control of quality of coal purchased by the Army, Air Force, and other Government agencies from Alaska mines. In 1958, tipple samples and a face sample were collected at three coal mines submitting bids for future military contracts. Periodic visits were made to Fort Richardson Army Base and Elmendorf, Eielson, and Ladd Air Force Bases in the Anchorage and Fairbanks areas to inspect sampling facilities, to assist with coal sampling procedures and problems, and to train personnel in coal sampling. Four trainees received instructions in coal sampling and two were recommended for certification by the Air Command.

A total of 628 coal samples were received and analyzed at the Anchorage coal laboratory. The samples received primarily represented contract coal delivered to military bases.

Also in 1958 the coal analysis laboratory at Pittsburgh, Pa., analyzed approximately 48,000 samples of coal, coke, char, tar, coal-mine dust, and related substances. This number included more than 36,000 samples collected by Federal coal-mine inspectors during some 3,300 inspections in 22 states, (approximately double the number of samples in 1957).

The incombustible content was determined for all coal-mine dust samples to check conformity with the provisions of Public Law 552, Federal Coal-Mine Safety Act. Of the remaining samples, 9,400 represented government purchases on a penalty basis and tipple inspections; and about 2,200 were submitted by Bureau of Mines and other government organizations in connection with research and exploration projects on coal. Analysis of the 48,000 samples required almost 258,000 chemical and physical tests.

International Classification of Coal

Because of increased trade in coal among various nations following World War II, the Coal Committee of the Economic Commission for Europe in 1956 adopted an International Classification of Hard Coals by Type and recommended

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its use to the various governments. The application of this system to American coals was reviewed and the findings were published.8/

The term "hard coal" as used in the classification is based on European terminology and means coal with a gross calorific value of more than 10,260 B.t.u. per pound on the moist, ash-free basis. Coals classified in the American Society for Testing Materials (ASTM) system as anthracitic, bituminous, and higher-rank subbituminous are included in the international system according to this definition. The lower-rank subbituminous and lignitic coals are included in a separate brown coal/lignite classification system adopted recently by the Coal Committee of the Economic Commission for Europe.

According to the international system, hard coals are first divided into 10 classes according to volatile matter and calorific value. Although this division of coals into classes is somewhat similar to the ASTM classification, the international system further divides the classes into groups and subgroups according to caking and coking properties determined by prescribed bench-scale tests. Caking properties are determined by either the free-swelling or the Roga test in which the coal is heated rapidly. Coking properties are determined by either the Gray-King coke type test or Audibert-Arnu dilatometer test in which the coal is heated slowly. The coal type is designated by a three-figure code number in which the first figure indicates the class of coal according to volatile matter or calorific value; the second figure, the group according to coking properties.

As little information was available on coking properties of American coals by either of the prescribed coking tests, representative samples of bituminous coals of various ranks from nearly every coal-producing state were subjected to the Audibert-Arnu dilatometer test. In addition to the coking test, determinations of free-swelling index, calorific value, and proximate and ultimate analyses were made to provide data for the parameters required for classification of the coals.

According to results of the survey, American coals fit into the international system as shown in Figure 1. American coals occur in 26 of the 61 code numbers as shown by the open-faced blocks. From the information available, there probably are no American coals that would be classified by the code numbers represented by the dotted blocks.

New Analytical Procedures

Many methods of analysis of coal and derived products have been developed by the Bureau and accepted as standards. However, to increase accuracy and efficiency, these methods are subject to continuous review, and new methods are being investigated.

A study was made of the effect of time elapsed between sample preparation and analysis on certain properties of coal. The effect of storage on four analytical samples of bituminous coals stored in open and closed containers

8/ Ode, W. H., and Frederic, W. H., The International Systems of Hard-Coal Classification and Their Application to American Coals: Bureau of Mines Rept. of Investigations 5435, 1958, 19 pp.

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GROUPS (determined by caking properties)			CODE NUMBERS										SUBGROUPS (determined by coking properties)		
GROUP NUMBER	ALTERNATIVE GROUP PARAMETERS		The first figure of the code number indicates the class of the coal, determined by volatile-matter content up to 33% V. M. and by calorific parameter above 33% V. M. The second figure indicates the group of coal, determined by caking properties. The third figure indicates the subgroup, determined by coking properties.										SUBGROUP NUMBER	ALTERNATIVE SUBGROUP PARAMETERS	
	Free-swelling index (crucible-swelling number)	Roga index												Dilatometer	Gray-King
3	>4	>45					435	535	635				5	>140	$>G_8$
						334	434	534	634	734			4	>50–140	G_5–G_8
						333	433	533	633	733			3	>0–50	G_1–G_4
						332a / 332b	432	532	632	732	832		2	⩽0	E–G
2	$2\frac{1}{2}$–4	>20–45				323	423	523	623	723	823		3	>0–50	G_1–G_4
						322	422	522	622	722	822		2	⩽0	E–G
						321	421	521	621	721	821		1	Contraction only	B–D
1	1–2	>5–20			212	312	412	512	612	712	812		2	⩽0	E–G
					211	311	411	511	611	711	811		1	Contraction only	B–D
0	0–$\frac{1}{2}$	0–5	000	100 (A / B)	200	300	400	500	600	700	800	900	0	Nonsoftening	A
CLASS NUMBER →			0	1	2	3	4	5	6	7	8	9	As an indication, the following classes have an approximate volatile-matter content of: Class 6 33–41% volatile matter; 7 33–44% " "; 8 35–50% " "; 9 42–50% " "		
CLASS PARAMETERS: Volatile matter (dry, ash-free) →			0–3	>3–10 (>3–6.5 / >6.5–10)	>10–14	>14–20	>20–28	>28–33	>33	>33	>33	>33			
CLASS PARAMETERS: Calorific parameter [a] →			–	–	–	–	–	–	>13,950	>12,960–13,950	>10,980–12,960	>10,260–10,980			
CLASSES (Determined by volatile matter up to 33% V. M. and by calorific parameter above 33% V. M.)															

Note: (i) Where the ash content of coal is too high to allow classification according to the present systems, it must be reduced by laboratory float-and-sink method (or any other appropriate means). The specific gravity selected for flotation should allow a maximum yield of coal with 5 to 10 percent of ash.
(ii) 332a . . . >14–16% V. M.
332b . . . >16–20% V. M.
(iii) From information available, American coals are represented by open-faced blocks. There are probably no commercial deposits of American coals represented by ·:·:·:·

[a] Gross calorific value on moist, ash-free basis (30° C., 96% relative humidity) B. t. u./ lb.

FIGURE 1. - Grouping of American Coals in International System of Classification.

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was evaluated.[9] Coals from Pennsylvania, Indiana, and Colorado showed only slight decreases in heating value after two months' storage, while a significant change was noted in an Ohio coal. The free-swelling indexes for the Pennsylvania, Ohio, and Colorado coals did not change during the five-month period of observation, but the Indiana coal changed one point at the end of four months.

Coal Ash and Mineral Matter

Results of analyses of a series of coals, ashed by both ASTM and International Organization for Standardization (ISO) procedures, showed that the heating rate of the proposed ISO standard method was too rapid. A second series of samples was analyzed and results submitted to the chairman of Working Group 2, ISO. These values were accepted and resulted in a modification of the procedure proposed in the ISO standard. The rate of heating determines to a great extent the fixation of sulfur in the ash; the faster rate of heating fixed more of the sulfur in the ash.

During this study, other factors were observed to influence the results of coal-ash determinations. The rate of heating, ventilation, location of crucible in furnace, and thickness of sample in the crucible are all vital factors. Several tests also indicated that the manner in which the coal is placed in the crucible affects the results.

Classical procedures for analysis of coal ash require time-consuming chemical separations of each of the elements. In recent years, a number of physicochemical techniques which largely eliminate the necessity of lengthy chemical separations have found considerable application in analytical chemistry. An investigation is in progress to determine the application of such methods to the analysis of coal ash. Tests of spectro-photometric methods for the colorimetric determination of iron, aluminum, titanium, and vanadium in coal ash showed that the methods are satisfactory. Although a separation to remove iron is required in the determination of microgram quantities of vanadium, the other elements can be determined directly in a solution of the ash with a minimum of manipulation. Preliminary investigation of a titration procedure for calcium and magnesium indicates that it probably is accurate enough for most purposes. The flame photometric procedure for sodium and potassium investigated earlier was found to be especially useful, particularly in connection with studies of the mechanism of the formation of deposits on superheater surfaces of boilers where only small quantities of samples are available for analysis.

An auxiliary program has involved the determination of germanium in coal ash from power plants.[10]

9/ Abernethy, R. F., and Tarpley, E. C., Change in Calorific Value and Certain Other Properties of High-Volatile Bituminous-Coal Samples During Storage: Bureau of Mines Rept. of Investigations 5386, January 1958, 13 pp.

10/ Corey, R. C., Myers, I. W., Germanium in Coal Ash From Power Plants: Coal Utilization, vol. 12, No. 11, November 1958, pp. 33-35.

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Empirical formulas are generally used to convert the ash content of coal to mineral matter content, which is needed for calculation of coal analysis to a dry, mineral matter-free basis. Two methods for direct determination of mineral matter in coal are being evaluated. Investigation of an acid-extraction procedure developed in Germany showed that with slight modification, (a hydrochloric acid pretreatment to remove calcium compounds) the method was satisfactory for all ranks of American coals. After this preliminary treatment, the bulk of the mineral matter is removed by treatment with hydrofluoric and hydrochloric acids as prescribed in the original method. Mineral matter is determined from loss in weight of the coal sample by the acid treatment, with corrections for the weight of residual ash, pyrite not removed, and chlorine taken up by the coal. Nineteen American coals ranging in rank from anthracite to lignite were tested by the acid-extraction method and the results compared with values calculated by empirical formulas. The precision was satisfactory and the results probably were closer to the true mineral matter content than those obtained by empirical formulas.

Empirical formulas may be criticized because the average factor for correction for the water of constitution of the shaley material may be in error for a particular coal. This objection is eliminated by the acid-extraction method because removal of the shaley material reduces to a minimum any errors that may result from applying average factors to the small amount of ash remaining after the acid treatment.

Unsuccessful attempts were made to determine qualitatively and quantitatively the mineral components of coal after dissolving the organic coal substance. Of several ranks of coal treated with abietic acid as solvent, a high volatile B bituminous coal had the highest solubility--97 percent on a dry, ash-free basis. Solubilities decreased for both higher and lower rank coals. Certain petrographic components, such as fusinite, micrinite, and spore exinite, were relatively insoluble in abietic acid, whereas vitrinite, particularly that of high volatile bituminous coal, was quite soluble.

Chlorine

The oxygen bomb-combustion method for determining chlorine in coal was modified by mixing one gram of Eschka mixture with the coal sample. Apparently this slows down the combustion, and unburned particles are not ejected from the crucible. This modification was used by Strambi in his proposal for total sulfur in coal.

A project was inaugurated in February 1956 to collect information on the chlorine content of United States coals from samples collected by Bureau of Mines engineers at the tipple. The results of 395 samples obtained from 14 states indicated that the average chlorine content ranged from approximately .01 to .13%.

Mine Dust

Most of the coal-mine dusts consist of coal and limestone. When slowly heated at 500° C. in air, the coal is burned, its ash remains, and the lime-

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stone is only slightly decomposed. Tests were made to accelerate burning of coal and repress dissociation of limestone by replacing air with a mixture of oxygen and carbon dioxide. If dissociation of the limestone can be avoided, a separate determination of carbon dioxide can be eliminated. A series of tests at 500° C. indicated that 3 hours are required to ash the coal. Investigations at 600° and 700° C. are planned.

Structure and Chemistry of Coal

Fundamental physical and chemical research on the constitution of coals and related materials was continued. A knowledge of coal structure would permit its better utilization as a source of liquid fuels, chemicals, and other carbonaceous materials. The varied applications of physical and chemical methods during the last 15 years have led to several tentative suggestions regarding coal structure. However, data collected to date are not sufficient, and the structure of coal still remains a mystery. Attempts are therefore being made to obtain additional data with more refined physical techniques and by a variety of new approaches and methods.

A large portion of the research program on coal constitution has been directed to the application of the principles of modern solid-state physics. Studies in progress on the optical properties of coal were integrated with a new approach to the problem of molecular structure of coal based on reflectance spectra and light scattering.

Indexes of Refraction and Absorption

The optical differences of components of coal under the microscope have long served as a basis for characterizing and differentiating parts of coal that behave differently when heated. Basic optical properties of coal were therefore determined not only for their value in revealing the fundamental structure of coal, but also in establishing an objective basis for characterizing the components of coal that have different thermal reactivities.

One phase of this investigation of the optical properties of coals and their components has been completed.[11/] Maximum and minimum indexes of refraction and absorption of vitrinites of different rank and of single crystals of graphite were determined from their microscopic reflectivities in different media. For the coal-graphite series, a relatively simple relationship was found between both reflectivity and the index of absorption and the hydrogen-carbon ratio, a measure of coalification.

Spectroscopic Studies

Ultraviolet and visible absorption spectra of Bruceton vitrain and similar data for naphthalene and higher polynuclear condensed aromatics were used to determine the possible maximum content of polynuclear condensed aromatics in the vitrain. The results show that the maximum content is lower than has been

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11/ McCartney, J. T., and Ergun, Sabri, Optical Properties of Graphite and Coal: Fuel, vol. 37, No. 3, July 1958, pp. 272-282.

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generally believed. Differences in the spectral intensities of Pittsburgh vitrain and a large number of 5- and 6-ring polynuclear condensed aromatics support this conclusion.

The color of coal may be attributed to free radicals. Recent measurements of the free radical content of lignite and Bruceton vitrains have shown an improved correlation between free radical content and ultraviolet extinction coefficients.

Studies on coal hydrogenation asphaltene in solvents have also shown a quantitative correlation between color and free-radical concentrations, both increasing with increasing solvation.

The intense ultraviolet-visible absorption observed in coal and in metal-carbonyl complexes was thought to be due to charge-transfer complexes involving electron transfer, but studies of the spectra of solutions of coal asphaltene and of metal-carbonyl complexes as a function of concentration have shown that these materials are not charge-transfer complexes. The infrared spectrum of the nitrobenzene-aniline complex was investigated to obtain information on the vibrational optical properties of such complexes.12/

X-ray Diffraction

Structural analysis of coal by x-ray diffraction requires a knowledge of the angular intensity distribution of scattered x-rays; a correction must be applied for absorption of x-rays by the coal specimen. A new method was developed for precisely determining x-ray absorption by inhomogeneous materials.13/ This method is also applicable to absorption of gamma rays, neutrons, and visible light.

Information concerning the structure of coal can also be obtained by subjecting it to mild thermal or chemical reactions. One process involves heating under vacuum and immediate condensation of the volatilized products. An x-ray study of vitrinite from high volatile A bituminous coal and of distillate and residue prepared from it by high-vacuum pyrolysis indicated that in the vitrinite the planar aromatic nuclei were small, consisting of one to three rings and that appreciable quantities of aliphatic peripheral groups were not present; that is, the vitrinite is composed of mainly cyclic structures.14/ Reaction of coal with lithium in ethylenediamine, (See also paragraph under Mild Chemical Treatment.) decreased the free-radical content of coal considerably. To determine the effect of the reduction on the carbon skeleton of coal, an x-ray investigation was made.15/ The major change in the

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12/ Friedel, R. A., Infrared Spectrum of the Nitrobenzene-Aniline Molecular Complex: Jour. Phys. Chem., vol. 62, No. 10, October 1958, pp. 1341-42.

13/ Ergun, Sabri, and Tiensuu, V. H., Determination of X-Ray Absorption Coefficients of Inhomogeneous Materials: Jour. Appl. Phys., vol. 29, No. 6, June 1958, pp. 946-949.

14/ Ergun, Sabri, Graphitelike Layers in Coals and High Vacuum Distillation Products: Fuel, vol. 37, No. 4, October 1958, pp. 365-370.

15/ Ergun, Sabri, and Wender, Irving, X-Ray Scattering Intensities of Anthraxylons Reduced with Lithium in Ethylenediamine: Fuel, vol. 37, No. 4, October 1958, pp. 503-506.

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x-ray diagrams occurred in the distance between planes which was increased by the addition of hydrogen from approximately 3.6 A. to 4.8 A. In reduced coals, carbon atoms in the hydrogenated layers are displaced from planar positions to form buckled layers.

Petrographic Studies

The thermal behavior of petrographic components of coals was studied by determining times required for fusion and resolidification at constant temperatures in the range 450° to 500° C. using a hot-stage microscope. Data collected on exinites permitted a systematic kinetic analysis. Vitrinites from two high-volatile bituminous coal were studied in a narrower temperature range, 464°-490°C. Reactions of micrinite and fusinite could not be studied quantitatively under the microscope. Qualitative observations showed that exinites and vitrinites heated at slow rates behaved different than those heated instantaneously, as in the present study. Under the former conditions, the data on fusion, especially of vitrinites, suggest that depolymerization or cracking is the initial step of thermal decomposition.

Concentrates of petrographic components of a high volatile A bituminous coal were obtained by centrifugal separation in salt solutions. Samples containing 94 percent vitrinite, 86 percent exinite, 87 percent micrinite, and 96 percent fusinite were obtained. Various physical and chemical properties of the concentrates were determined; for example, chemical composition, helium density, surface area, indices of refraction and absorption, and x-ray scattering intensities. The components differed considerably in chemical composition and in most physical properties. Some of the physical properties could be related to chemical composition.

Mild Chemical Treatment

When metallic lithium is dropped into a solution or suspension of an organic substance in ethylenediamine at 90°-100°C., unsaturated bonds in the organic material are extensively hydrogenated. Coals, vitrains of various ranks, and even graphite have been reduced by lithium in ethylenediamine.[16 17/] Since the carbon skeleton of the coal is undisturbed by the treatment, this method should provide much-needed information on the constitution of coal.

The amount of hydrogen added reaches a maximum when the carbon content is approximately 90 percent; for this carbon content, about 44 atoms of hydrogen are added for every 100 carbon atoms. It is difficult to hydrogenate coals of such high rank catalytically.

16/ Reggel, L., Raymond, R., Friedman, S., Friedel, R. A., and Wender, I., Reduction of Coal by Lithium-Ethylenediamine: Fuel, vol. 37, No. 1, January 1958, pp. 126-128.

17/ Reggel, Leslie, Friedman, Sidney, and Wender, Irving, The Lithium-Ethylenediamine System. II. Isomerization of Olefins and Dehydrogenation of Cyclic Dienes: Jour. Org. Chem., vol. 23, No. 8, August 1958, pp. 1136-1139.

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The solubilities of the coals in pyridine at room temperature were studied before and after such treatment. Coals of 89-90 percent carbon have limited solubility, as the molecules are large and highly aromatic. On reduction, considerable conversion to more soluble hydroaromatic structures takes place, and as much as 90 percent of this coal becomes soluble in pyridine. The platelet size of a 91-percent carbon coal is so large that even the highly reduced hydroaromatic type of structures have limited solubility.

Treatment of higher rank coals with lithium in ethylenediamine results in the elimination of over 85 percent of the sulfur originally present; about two-thirds of the sulfur in lower rank coals is eliminated by this treatment.

Both hydrogen and carbon monoxide are added to coal when it is treated with synthesis gas at 200°C. in the presence of dicobalt octacarbonyl. Hydrogen adds (1) by saturating aromatic rings in polynuclear hydrocarbons (polycyclics are hydrogenated to molecules containing isolated benzene rings or phenanthrene nuclei), and (2) by reacting in concert with CO on double bonds (hydroformylation) to form primary alcohols. As much as 44 percent of Rock Springs, Wyo. coal is soluble in phenol after this treatment. Only 19 percent is soluble when hydrogen alone is used, and 12 percent when helium is used. Experiments at higher temperatures such as 250°C. with m-cresol show that as much as 67 percent of the coal is solubilized in the catalyzed reaction as compared with only 19 percent in the absence of a catalyst.

Tests with radioactive carbon monoxide showed that approximately 1 molecule of CO was added for every 200 carbon atoms in an 83-percent carbon coal; approximately 1 molecule of CO was added for every 40 carbon atoms in a low rank (68 percent carbon) coal. The addition of carbon monoxide is good evidence for the presence of nonaromatic double bonds in coal. On the assumption that indene-type structures may be responsible for this reaction, both indene and acenaphthylene were treated with sythesis gas and dicobalt octacarbonyl; the chief reaction was saturation of one double bond to form an indane type of structure, but some addition of CO (5-20 percent) occurred. The acenaphthylene reaction is being studied as a direct means of determining the amount of indene structures in coal.

Chemical Treatment of Anthracite

Little information is available on the fundamental chemical characteristics of anthracite. Samples of anthracite have been treated under reflux with concentrated nitric acid for periods up to 1,000 hours. The acid-soluble and acid-insoluble portions of the product have been separated, and studies conducted on both to attempt to isolate any chemical entities that have been formed and to determine the characteristics of the reaction products. The proportion of original carbon converted to acid-soluble products ranged from 0.2 percent after 24 hours to 17.6 percent after 1,000 hours. Products recovered from the acid-soluble fractions vary from 4.6 percent of the original coal weight after 24 hours oxidation to 41.3 percent at the 1,000-hour period. The ash content of the acid-soluble oxidation products decreased from 32.9 at 24 hours to 10.3 at 1,000 hours. The amount of 5 percent sodium hydroxide soluble in the oxidation residues increased from 4.9 percent at 24 hours to

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18.5 percent at 192 hours. This characteristic remains essentially constant throughout subsequent stages of oxidation up to, and including, 1,000 hours. Attempts to characterize the residue by solvent extraction have been only partially successful. The solvent action of an organic base, such as dimethylformamide, disperses the oxidized residue almost as effectively as aqueous alkali. Attempts to separate individual components from the oxidation products by chromatography have not been successful. Some evidence of separation has been noted in columns packed with silica gel, and also in others packed with sucrose, but in both cases the bands have not been clearly defined. Tests based on electrofluoresis have been conducted. However, no definite conclusions can be drawn from them.

Functional Groups in Coal

The reaction of hexamethyldisilazane, $(CH_3)_3SiNHSi(CH_3)$, and trimethylchlorosilane, $(CH_3)_3SiCl$, with coal has been developed into a method for determining hydroxyl groups in coals. Its two advantages over previous methods are the virtual freedom from side reactions and the fact that even greatly hindered hydroxyl groups react. As a consequence, this method is being used to check the reliability of older means of determining hydroxyl groups in coals. The effects of concentration of reagents, time of reaction, and presence of air on the hydroxyl values have been studied. A series of eight vitrains have been analyzed by the trimethylsilyl ether method and their hydroxyl contents determined as a function of coal rank.

In this connection, the catalytic hydrogenation of trimethylsilyl ethers of alcohols and phenols has been investigated. Raney nickel catalyzes the hydrogenolysis of the carbon-oxygen bond in secondary alcohols and in phenols at comparatively low temperatures and may provide a means of selectively removing hydroxylic oxygen from coal.

Humic Acids

Humic acids are readily obtained from naturally or artificially oxidized lignite. Ultimate utilization of the acids as such or as intermediates, and their use in solving problems of coal structure and coalification may result from current investigations. Study of the humic acids obtained from naturally oxidized lignite (leonardite) was continued with preparation of 200- to 300-gram samples from materials obtained in two widely separated locations. Sample preparation consists of extraction with alkali, centrifuging to remove insoluble matter, precipitation by acidifying the caustic solution, and then collecting the acids and washing exhaustively with large volumes of water. Extraction of these humic acids with alcohol produced a material of higher ethoxy content and similarly extraction with methanol yielded higher methoxyl content, indicating the acids are easily esterified. Small amounts of wax (alcohol-benzene soluble) are also recovered. Humic acids as they occur in leonardite are not extractable to any great extent with organic solvents, but a light acid treatment sharply increases the extractability with acetone-water solutions. This effect is being investigated further.

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Microbial Degradation

Studies of the bacteriostatic and fungistatic materials in coal have indicated that preparations obtained by solvent extraction are active at a concentration of 1:200,000, approaching within a factor of less than 10 the activity of penicillin under the same test conditions. The sodium hydroxide-soluble fraction of the methanol extract of coal had the greatest activity. Testing of this fraction by a pharmaceutical laboratory has shown at least one antibiotic component to be present; however, the amount of sample was insufficient for characterization of the active principle. Attempts are being made to increase both yield and potency of the active materials by steam distillation of the whole coal.

Experiments with fungi growing on an Illinois coal showed that growth was stimulated when coal and a supplemental carbon source (glucose) were provided and exceeded growth on glucose alone. Growth was not only proportional to the amount of coal provided, but seemed more vigorous on certain methanol-extracted samples. Experiments in percolation units with coal as a sole source of carbon and soil as inoculum showed rapid increase in bacterial numbers in the first four days followed by a rapid decline. Bacteria isolated from these percolators were pseudomonads.

Isolation of bacteria capable of using combined organic sulfur or nitrogen is in progress. Studies on the oxidation of polycyclic aromatic compounds by bacteria were continued.

A steric effect of a methyl group in microbial oxidation of aromatic ring compounds has been found. The presence of a methyl group adjacent to a hydroxyl has a definite hindering effect. Another steric effect has been noted with bacteria induced to oxidize quinic acid; these were able to grow on salicylic acid and p-hydroxybenzoic acid, but were unable to attack the isomeric m-hydroxybenzoic acid.18/

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Irradiation of Coal

In response to widespread interest in the effects of high-intensity nuclear radiation on coal, a program was initiated to study the effects of irradiation with gamma rays (gamma irradiation) and neutron plus gamma rays (reactor irradiation): (1) The Bureau of Mines will have coal samples irradiated at the highest gamma and neutron flux-levels available: (2) Samples of irradiated and unirradiated coal will be subjected to standard tests, and changes in coal structure, composition, or properties due to irradiation will

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19/ Ezekiel, W. N., Society for Industrial Microbiology, Washington Section (P.2). Science, vol. 127, No. 3295, Feb. 21, 1958, pp. 428-429.

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be determined. Similar studies will be made of samples of irradiated coal received from other organizations.

Irradiated samples will be subjected first to "non destructive" tests—size reduction, electrical conductivity, optical properties, density (helium method), and surface area (nitrogen adsorption method)—and will be subsequently re-used for "destructive" tests—density (mercury method), plasticity, sorption, solubility, ultimate analysis and functional group analysis.

The Engineering Test Reactor (ETR) at Idaho Falls, Ida. is the only facility available that can provide a flux of 10^{15} neutrons per sq. cm. per second, and will be used initially for the irradiation of a sample of Rock Springs coal weighing about 7 grams, with ash reduced by hand sorting to approximately 1 percent (to keep induced radiation to a minimum), in a container having six chambers, each holding a column of 40-mesh coal. The container is designed so that the gamma-induced temperature at the center of a coal column will not exceed 400° F. Metallic (bismuth and lead) and organic temperature-measuring devices were embedded in the coal columns. The sample container was evacuated, filled with helium to 1 atmosphere pressure, sealed, and sent to ETR. A duplicate "blank" sample was held for later comparison with the irradiated sample. Subsequently 15-gram samples of each of the two coals will be irradiated—Rock Springs (high-volatile bituminous C) and Pocahontas (low-volatile bituminous). If these test results show that significant changes in the coal have taken place, a program will be inaugurated to investigate the use of nuclear radiation in coal processing.

Fundamental research on the effect of radiation on anthracite was also inaugurated. Low, medium, and high-volatile (about 4, 5, and 7 percent, respectively) anthracites have been exposed to integrated neutron fluxes of 10^{18}, 10^{16}, and 10^{14} nvt. (n=neutrons per unit volume of beam, v=neutron velocity, t=exposure time) and to cobalt-60 gamma dosages of 10^{8}, 10^{7}, and 10^{6} rads (one rad equals 100 ergs of radiation per gram). Mixtures of medium-volatile anthracite with elemental sulfur or with calcium oxide in a 1:1 ratio have been exposed to the maximum available neutron flux and maximum gamma dosage. Each exposure was made on 50-gram samples of minus-60- plus 140-mesh anthracite. Tests are being conducted to determine the analyses of the gases evolved during radiation, and the effects of radiation on the porosity, grindability, ease of oxidation, and electrical conductivity of the samples.

The Federal Geological Survey has sponsored irradiations of humic acid and lignite in the Materials Testing Reactor (MTR) at Idaho Falls—irradiations performed at fluxes up to 4 x 10^{14} neutrons per sq. cm. per second. A 2-pound sample of Pittsburgh-seam coal (high-volatile bituminous) from Ohio was gamma-irradiated for the Consolidated Coal Co. in the Argonne National Laboratory, Lemont, Ill.—at a flux of 10^{7} roentgens per hour for 30 hours. Samples of these irradiated materials and corresponding unirradiated blank samples are being made available to the Bureau for analysis.

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EXPLORATION AND MINING

Exploration of Alaskan Coals

The Bureau of Mines is cooperating with other Federal agencies in establishing the extent and value of coal deposits in Alaska, and in conducting research to determine the most efficient and economic methods of mining and utilization. Since Army and Air Force installations in the vicinity of Anchorage and Fairbanks depend on coal as a source of heat and power, attention has been directed towards the development of adequate reserves and the establishment of privately operated facilities capable of maintaining an uninterrupted supply of solid fuel for both military and civilian requirements.

The overall program includes geological investigations, exploration, and development of the potentially large coal deposits, the devising of mining procedures for safer and more economic recovery of the coal, beneficiation, and other studies toward the full use of the heat, power, and by-product potential of the resource.

The Bureau of Mines conducts (1) reconnaissance examinations of coal deposits by trenching and diamond core drilling, (2) laboratory studies to indicate methods of up-grading the coals, and (3) supervision of sampling procedures and analyzing samples to assure the proper quality and prompt settlement of coal deliveries to military and other Government establishments.

Nearly all of the core drilling to date has been in the Matanuska coalfield, which produces the highest rank coal currently being mined in Alaska. Drilling is done in close cooperation with the Geological Survey and supplements previous mapping or preliminary exploration by that agency. Core drilling operations in the Moose Creek area of the Matanuska coalfield were continued in an attempt to obtain data on the location, extent, and character of the Jonesville and Premier series of coal measures in the unexplored sections of the south limb of the Wishbone Hill syncline. Earlier drilling explorations indicated the possible existence of geological complications in at least some parts of the Moose Creek area that would adversely affect both reserves and mining conditions. New attempts were made to investigate these conditions further and to obtain critical reserve data. Penetration was attempted of both the Jonesville and Premier series at a point near the projected position of the synclinal axis.

The evidence obtained from the recent trials indicates clearly that a major fault (perhaps the Buffalo fault) has influenced the position of the coal measures (and possibly the position of the synclinal axis) in this immediate area, and the mechanical difficulties involved in drilling this area make additional investigations impractical.

Additional work begun during the year included the acquisition and assembly of special equipment to be used in the proposed investigation of the Beluga River coalfield. A preliminary investigation of this field indicated

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favorable possibilities for the development of large reserves of strip coal that might be used for the production of on-site power for transmission to Anchorage.20/

Anthracite Mining

The competitive position of anthracite in the fuel markets of the United States undoubtedly would be improved by a substantial reduction in its price. Because mining expense represents such a large share of the f.o.b. mine price, the Bureau's mining research has been directed largely toward the development of low-cost, highly-mechanized mining systems for underground operations. In efforts to overcome the difficulties of mining moderately and steeply pitching coal beds and to adapt high-productivity coal-mining machinery to these conditions, the development of low-cost longwall systems, and the modification of current mining systems to permit increased mechanization are being investigated. Basic to the successful application of such systems, new methods of roof-control are also being studied to assure improvement in the safety of the workers and increased coal recovery.

Longwall Mining

The longwall mining investigation is divided into three phases: (1) Establishing a longwall face by undercutting with conventional machines, blasting, and loading the coal with a rigid-blade planer; (2) experimenting with continuous mining machines, a drum-cutter loader and the Bureau's vibrating blade planer; and (3) mechanizing the roof-support system (fig. 2).

During the short time of testing equipment in phase 1, about 13,000 tons at a productivity rate of approximately 5.1 tons per man-shift, and loading rates of 75-90 tons per hour were obtained with the rigid-blade planer. However, with a strong sandstone roof the coal overhang had to be blasted into the open area (approximately 20 feet) between the coal face and the cave line.

Phase 2 will begin with the installation of the drum-cutter loader. Following thorough tests of this equipment, the Bureau's vibrating-blade planer, which has been rebuilt, will be installed and tested. The maximum open roof area (solid coal to cave line) to be supported will be reduced to 11 feet by eliminating the conventional undercut and shifting the cave-line props nearer the solid coal face. In addition, prop concentration will be increased approximately one-third.

Mechanization in Pitching Beds

A cooperative agreement was made to test and develop mechanized mining suitable for beds with a pitch of more than 20°. The Mammoth bed in the Western Middle field, a 30-foot thick seam with a pitch of 20° to 45°, is being used for the tests. Gangways of 300 feet are driven with a continuous miner of the borer-trimmer type, leaving a 35-foot pillar between roads. (See

20/ Maloney, R. P., Reconnaissance of the Beluga River Coalfield, Alaska: Bureau of Mines Rept. of Investigations 5430, 1958, 18 pp.

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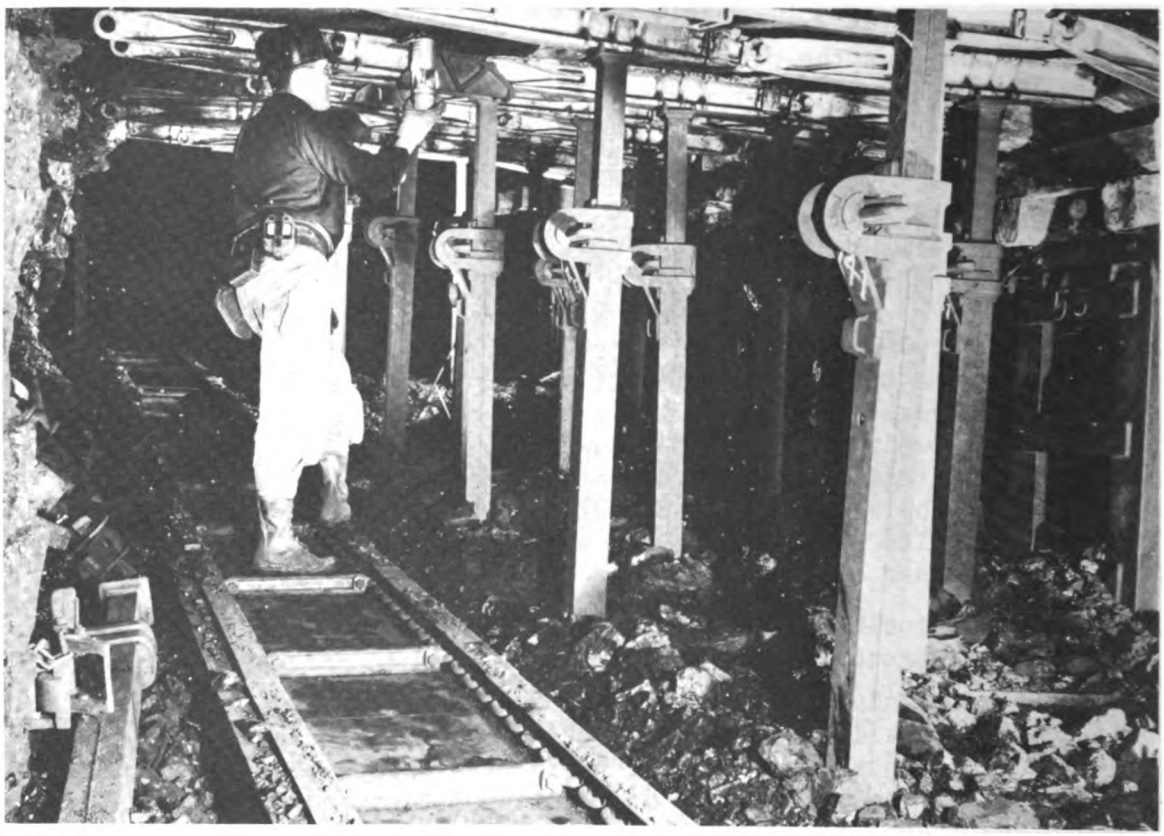

FIGURE 2. - Armored Chain Conveyor Along Face and Roof-Support System, Showing Yielding Steel Props, Aluminum-Alloy Bars, Bureau-Designed Steel Crib Bases, and Crib Releases.

fig. 3.) The borer is then withdrawn and moved to a new location down the pitch. Holes are drilled with a 2-foot auger equipped with a 4-foot reamer from the completed gangway to the projected gangway below. These auger holes serve to bleed the gas from the coal ahead of the projected advancing gangway, as escapeways, and ventilation holes. After augering is completed, mobile loaders and conveyors are placed in the roadway and the pillars are mined. The 8 by 12 ft. borer section is supported by four-piece yielding arch steel timber made from heavy-duty steel mine tie sections. Preliminary results indicate that production could average 30 tons per man-shift. Longer gangways would contribute to the realization of this objective, and it is hoped that eventually gangways can be driven up to several thousand feet in length. However, before this can be accomplished the borer must be adapted to use on soft bottoms or restricted to areas where a firm floor is present. Meanwhile, a rock slope is being driven that will permit gangways of 500 to 800 feet in length.

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FIGURE 3. - Pitch-Mechanization Project: Completed Borer-Driven Coal Gangway Showing Yielding, Arch Type Steel Roof Support.

Hydraulic Hoisting

A research project on hydraulic hoisting of anthracite was initiated. Tests were made to obtain basic information on the settling of solids of various sizes, shapes, and specific gravities in a column of water. A vertical, transparent tube 6 inches in diameter and 50 feet long is now used for test purposes. Results of almost 1,000 tests indicated that the greatest factor in sinking speed is the shape of the specimen. Spherical and cubical shapes have higher sinking velocities than flat "A" and oblong pieces.

With the conclusion of the sinking velocity tests, a pipe column will be connected to a centrifugal pump so that actual pumping tests can be made at different velocities to determine rising velocities and the amount of degradation incurred in passing coal through the column. For test purposes, a variable-speed centrifugal pump was procured, which has an impeller that will permit the passage of solids up to 1-3/4 inches (Nut size). A preliminary design was made of a lock-chamber feed apparatus, for automatically feeding

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large-size solids into a high-pressure pipeline. The design is sufficiently detailed to permit manufacture of a pilot-plant model.

Mine Water Control

A joint Federal-State program for the control of water in anthracite mines was initiated in 1955 under legislation passed by the Congress of the United States and the General Assembly of the Commonwealth of Pennsylvania. A sum of $17 million was provided for surface drainage installations, pumping works, and related facilities for the control and drainage of water to prevent the flooding of mine workings and the loss of anthracite reserves.

In 1958, five mine-water control projects with a contracted or estimated cost of nearly $2-1/2 million were approved. Two were pumping installations which required seven pumps with a total capacity of 28,000 gallons per minute. The remaining three were surface-drainage improvements designed to prevent about 750 million gallons of water from seeping into mine workings each year.

One pump and five surface-drainage improvement projects were completed in 1958 and are being operated and maintained by the sponsoring coal companies. (See fig. 4.) The pumping installation required two pumps with a total capacity of 10,000 gallons per minute. The five surface-improvement projects involved backfilling old strip pits and crop falls, constructing concrete flume and pipe, steel flume and pipe, railroad culverts and other surface work. It was estimated these completed surface projects will keep out of the mines more than 800 million gallons of surface water each year.

At the end of 1958, a total of 18 projects were either in an active status or had been completed. The contracted or estimated cost of these projects totals nearly $6-3/4 million, an amount shared equally by the Federal and State governments. In addition to the six projects completed in 1958, one surface-drainage improvement was completed in 1957.

Bituminous Coal Mining

The objective of the work on bituminous coal is to develop new or improved mining methods that will increase productivity, coal recovery and therefore conservation, and safety for all mine workers. Throughout these investigations, data on various mining methods and the performance of equipment are collected, compiled, evaluated, and published as quickly as possible so that the results for given operating conditions can be compared and made available to the public.

Hydraulic Mining

Published reports on hydraulic mining of coal in foreign countries claim that this method is cheaper and safer than conventional mining in the same areas and that more complete recovery of coal can be made. As no company in this country has investigated this method, in spite of these favorable reports, the Bureau initiated a program to evaluate the application and efficiency of hydraulic mining. Some of the equipment necessary to conduct experiments has

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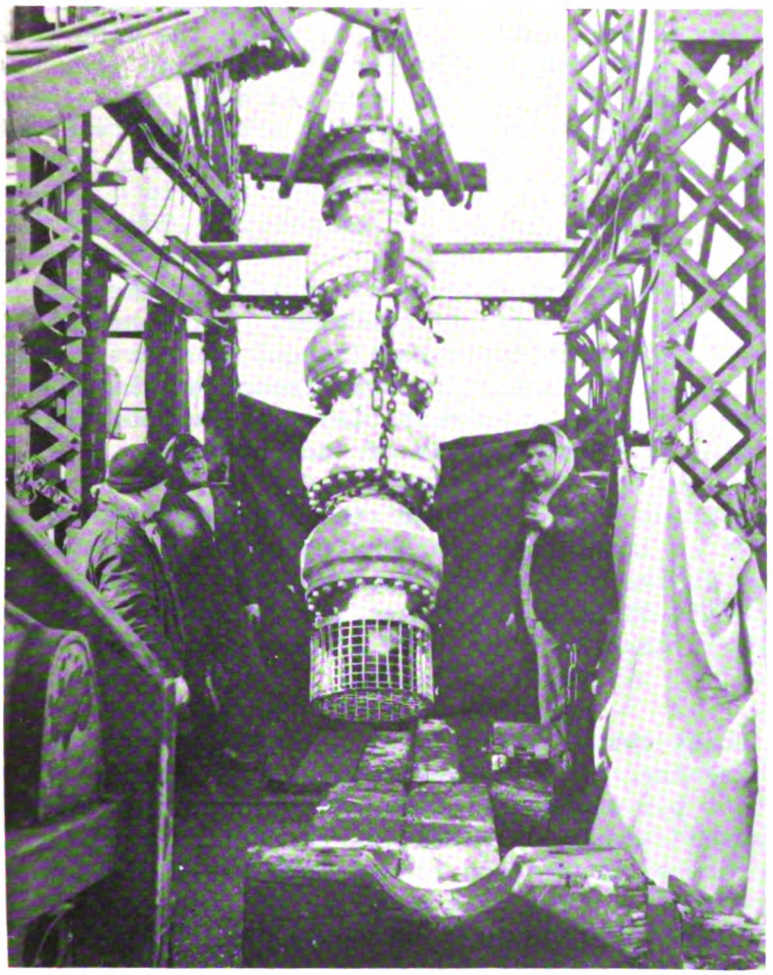

FIGURE 4. - Mine-water Control Program. Bowl Assembly of 5,000 g.p.m. Deep-Well Pump Before Lowering Into Shaft.

been assembled. A pump capable of delivering water at 300 g.p.m. at pressures of 4,000 p.s.i. was purchased, and 900-hp. diesel engine to drive the pump was obtained. Auxiliary starting and operating equipment has been ordered. Preparation of the experimental site has begun.

In the initial phase of the program, attempts will be made to find the maximum rate at which coal can be broken from a solid face. Information will be obtained to determine: The most effective shape and size of nozzle; the distance required between the nozzle and coal face for maximum production; the effect of water pressure and flow rate; the effect of cleavage planes in the coal on rate of production; and the size consist of the coal mined. In addition, the effect of water on the floor, ribs, and roof of the openings, will be determined, and a method for collecting and disposing of waste water will be developed.

Roof Bolting

Roof control is still a major mining problem. Roof falls are the chief cause of injuries and increase production costs. Although the initial cost of supporting a mine roof with bolts is higher, roof bolting is more efficient than timber support. However, a study made in 14 mines proves that, in addition to safety considerations, the use of bolts offers other advantages which generally compensate for higher initial costs. In highly mechanized mining, especially when continuous mining machines are used, the roof can be bolted with minimum stoppage of loading, thereby increasing the production time. The need for costly clean-up of falls and retimbering has been reduced more than 75 percent when bolts are used.

At seven mines productivity in bolted sections was from 2 to 7 tons per man-shift more than in timbered sections. In addition, the use of roof bolts

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does not always result in a direct increase in the cost of roof support—this cost decreased 1 to 5 cents per ton at five of these mines, although it increased 1 to 6 cents per ton at the other two mines. Changing from timber to roof-bolt support may be impractical and unnecessary where the mine roof is strong and can be supported with posts only. The benefits derived from roof bolting were greatest where the immediate roof stratum was weak and fragile; and in some cases the use of bolts permitted mining that could not be done economically with timber supports.

Face Haulage

Face haulage is the first step in the process of moving systems and equipment for mined coal from the working face out of the mine. In mechanized mining, where working places and high productive capacity equipment are concentrated in a few sections of a mine, it is particularly important that mined coal be removed as quickly and efficiently as possible. The Bureau, therefore, undertook a program to determine the best systems of face haulage.

Results of studies at 12 mines have indicated clearly that good supervision and planning are at least as important in achieving productivity as the type of haulage system used. At one mine where cars are used in a 62-inch coal bed, a productivity of 37 to 51 tons per man at the face was obtained. At two other mines using cars in coal beds 72 to 92 inches thick, only 16 to 18 tons per man were produced.

In coal beds 36 inches thick chain conveyors with a bridge conveyor between the loading machine and the conveyor provide good face haulage for both continuous mining (20 tons per man-shift) and conventional equipment (16 tons). Extensible belt conveyors are used in beds 52 to 96 inches thick and seem to offer the best performance; production ranged from 33 to 77 tons per man-shift at the face. Where shuttle cars were used in coal beds from 52 to 96 inches thick, production ranged from 15 to 67 tons per man at the face.

Foreign Activities

The Bureau of Mines continues to participate in a program of technical assistance to friendly nations for the development of their solid fuel resources. This assistance is directed primarily toward the training of personnel in such countries and toward improving their technical competence in exploration, development, mining, and utilization. Bureau engineers are assigned to foreign technical assistance projects at the request of the International Cooperation Administration, and qualified foreign candidates are sent to the United States for advanced training at Bureau of Mines facilities.

Improved health and safety measures and training of native personnel for mining and related operations are problems of pressing concern to Bureau engineers assigned to posts in Afghanistan, Colombia, India, and Indonesia, where coal production must be increased to encourage industrial growth.

In all assistance programs, great emphasis is put on training in mine health and safety practices. In 1958 two Bureau health and safety teams

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conducted advanced training courses at several mines in Mexico. Over 11,000 copies of a Spanish translation of the Bureau's first-aid manual were printed in Mexico to fill requests received from Spanish-speaking countries.

Afghanistan

Bureau engineers have been cooperating in the development of Afghanistan's small coal industry since 1951. This country, with a severe winter climate and a population of approximately 12 million people, produced less than 30,000 tons of coal in 1958. Approximately 50 percent was used for domestic purposes and the remainder for power generation and in industrial plants.

The principal industrialized area is in the vicinity of Kabul, the capital. Coal resources exist in four areas; three are located approximately 300 miles or less from the capital. Except for a few hundred tons, two mines (the Ishpushta and Karkar Mines) accounted for all of the 1958 production. The Darra-Suf area, with reserves of subbituminous coal that can be measured in millions of tons, is still comparatively inaccessible and unproductive. All coal must be transported by truck and the only accessible roads are in the areas where the Ishpushta and Karkar coal mines are located. Although industrial growth trends in Afghanistan are very modest, fuel requirements are overtaking the combined production capacity of these two mines. If the government's efforts to stimulate the use of coal as a domestic fuel are successful and distribution facilities are improved, the increase in coal requirements will be greatly accelerated. Development of the Darra-Suf area was therefore a high priority objective in 1958. Construction of a road that would connect Darra-Suf to the Kabul approaches was begun. Opencut mining operations in the new area will begin in the spring of 1959 and continue until underground operations are planned and developed by the Bureau's engineer in cooperation with his Afghan counterparts.

The 104 American trucks purchased by the Ministry of Mines and Industry were delivered during the year and used to transport coal from Ishpushta and Karkar to Kabul. Part of this truck fleet will be available for transporting coal from the Darra-Suf area when production is started in that area.

Colombia

The outlook for a substantial and profitable coal industry in the Cauca Valley of Colombia continues to be favorable although progress was interrupted by a change in government and subsequent reorganization. A new government-sponsored company, Carbones del Valle y del Cauca, S.A., was organized with a capital of approximately $3 million, to assume management of the two largest producing mines in the area (Industria Hullera San Francisa, Ltda., and Hulleras de Timba, Ltda.), and the coal washing plant at Cali (Planta Lavadora). The two mines were closed for several months, but independent mine operators increased production to meet local demands. The Bureau's engineer assisted the small operators in revamping their mining systems to obtain greater production under improved safety conditions.

The mines along the slopes of the Cordillera Occidental, a range of mountains which parallels the Pacific coastline, are generally small producers

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relying on primitive mining methods—most of the coal is hand trammed; and only natural ventilation is available. These small, poorly-equipped units pose a problem in attempts to regulate coal production and quality for blending operations at the washing plant in Cali.

The washing plant, erected and operated by the Instituto de Fomento Industrial (a government corporation), buys coal from the mines in the district and prepares it for export. The plant was operated throughout the year, and in late September about 20,000 tons of washed coal was stored at the plant, and an additional 10,000 tons was on the dock at Buenaventura ready for export. The establishment of a regular export market for 10,000 to 20,000 tons of coal per month has long been one of the objectives of the Cauca Valley project. However, as the year ended, the coal at Buenaventura had not been sold and a reappraisal of export markets was being made.

The position of the washing plant was gradually strengthened by increased consumer demand for washed coal. Small mine owners were formerly reluctant to sell coal to the washing plant because the price (based on ash content) is usually lower than can be obtained on the open market. However, they are becoming increasingly aware that the plant represents their only sure and regular market and are now selling more of their product to the plant. American, French, German, and other foreign engineers inspected the areas around Cali, indicating that the coal resources of this region are of interest to foreign investors.

India

A pilot plant was erected in India for the Neyveli Lignite Corporation for producing carbonized smokeless briquets from lignite. After construction of the plant (fabricated by an American firm), test runs and final adjustments were made under the direction of Bureau engineers. The low-temperature carbonization process used in the Neyveli plant was developed by the Bureau of Mines. The government of India requested assistance in the adoption and application of this process to Indian conditions, to permit effective utilization of their large lignite deposits. Difficulties were encountered in the preliminary operation of the plant crushing and briquetting equipment because adequate quantities of the type of raw lignite for which the process was designed could not be obtained. However, these problems were resolved when a supply of normal run lignite was obtained, and the Neyveli plant is now in operation. This program was developed to provide a means of producing a smokeless, easily ignitable domestic fuel from raw lignite of very high inherent moisture content (50 to 60 percent). Design capacity of the crushing, briquetting, and drying units is 2.5 tons of raw lignite per hour; capacity of the carbonizer is 500 to 1,000 pounds per hour.

Indonesia

A variety of internal problems in 1958 impeded progress in efforts to improve the conditions that have contributed to a steady decline in Indonesia's coal production. The Bukit Asam mine in south Sumatra was reported to be the only mine that produced without serious interruptions, although its production

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in 1958, an estimated 502,000 tons, was below that of the previous year (about 555,000 tons). The Ombilin mine, the only other large producer in Sumatra, mined enough coal to meet only local demands. Coal mined in Kalimantan accumulated at three coaling stations in the area due to lack of transportation facilities, and several of the mines were closed due to a lack of funds. Coal production on the island of Java was negligible.

Operations at the Ombilin coal mines were investigated and a report was submitted by the Bureau's coal mining engineer assigned to International Cooperation Administration's (ICA) technical assistance project in Indonesia as an advisor to the Indonesian Bureau of Mines. The report incorporated recommendations for increasing underground mine production and opening a stripping operation in the C-Tanah Hitam area. Complete mining plans were prepared for an overall production of 1,500 tons per day from underground and strip operations.

The strip mining equipment purchased in 1955 has been operating since 1957 at the Bukit Asam mines near Palembang, in south Sumatra. Changes in operating procedures were recommended by the Bureau of Mines engineer to increase efficiency in removing overburden, and to produce cleaner coal. Improvements in maintenance of equipment were also suggested and a booklet on belt conveyor maintenance was prepared for the use of mine personnel.

A proposal made by the west Java War Administrator for development of the Bajab and Tjimandiri coal fields in west Java was evaluated. The deposits are lenticular, very irregular, and range from about 9 inches to 5 feet in thickness. More than 6 miles of railroad or highway would have to be constructed to move the coal from the mines to the consuming centers. These unfavorable conditions were brought to the attention of government officials.

Health and Safety

Roof Control

Research on problems of roof support, as characteristic of roof rock, providing temporary support to the exposed roof, and eliminating rock and coal outbursts, was intensified during the past year. The frequency of deaths and injuries from roof falls and the expanding use of mechanical equipment necessitated research of this type. Roof falls continue to be the biggest cause of fatalities in underground mining. Although major coal producers have been successful in reducing the frequency of these accidents, the number of accidents in small mines has increased where modern methods of roof control and support are not generally adopted and safety practices are not strictly enforced. Forty new mines adopted roof bolting as a means of support during the year, bringing the total number to 524.

Several methods have been successfully employed for recovering roof bolts in Indiana, Illinois, and western Kentucky coal mines.21/ Roof-bolt recovery

21/ Kelly, L. W., Roof-Bolt Recovery in the Middle West: Bureau of Mines Inf. Circ. 7826, 1958, 17 pp.

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and re-use is now an established practice in these states, and recovered bolts have been used in many mines over a period of several years without failure.

Roof bolts are sometimes bent to permit installation in areas where the height of the opening is less than the length of the bolts required to support the immediate roof adequately, and are also subsequently reclaimed and reused. A study was made to determine the effect of bending on the strength of bolts and whether the reuse of bent bolts is safe. The tests indicated that bent bolts can be reused a number of times if they are carefully inspected before each use, and are free of cracks or other deformities.22/ The bolts must be inspected before reuse for minute cracks within the bent portion of the rods. Routine inspection should begin when the roof bolt is straight. Provisions must be made to clean or keep the rod free of small particles of roof strata that would fill or camouflage any cracks that may be present. Mine officials may also decide on a limitation for reuse, by setting a limit on the maximum number of times a bolt can be bent and used before it cracks, becomes unsafe, and should be discarded.

Roof bolt specifications were prepared by a committee in which the Bureau participated, were approved in August 1957 by the American Standards Association, and presented for approval by the Roof Action Committee of the American Mining Congress.

A cooperative study is now being conducted with manufacturers to establish a standard anchorage testing procedure for expansion-type roof bolts. A standard method has been developed for determining the torque-tension relationship for any combination of roof bolting materials used.

A stratascope has been designed to explore the strata to a depth of 9 feet and can be used in boreholes having a diameter of 1-3/8 inches. The instrument is commercially available.

A project was initiated to explore the possibility of using sonic waves to probe and determine the stability of coal mine roofs. The feasibility of sending wave signals into and through samples of roof strata has been established. Laboratory studies of this technique are now in progress and will be followed by field experiments to compare the results obtained by sending and interpreting signals with known underground conditions.

Roof bonding, a process of consolidating layers of roof rock to increase strength by injecting a quick-setting cementing (or bonding) agent through drilled openings into the rock, is being studied. Experimental work has indicated that this process is feasible, and cost data have been obtained for certain conditions. Equipment has been designed to permit testing of this process in conjunction with actual commercial mining operations. Such equipment now is being assembled.

22/ Gaddy, F. L., Strength of Steel Roof Bolts After Repeated Bending: Bureau of Mines Rept. of Investigations 5408, 1958, 14 pp.

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Bumps or Coal Outbursts in Coal Mines

In August 1958 the Bureau began an investigation of bumps or coal outbursts in coal mines. Two widely separated localities where bumps occurred, the Pocahontas field in West Virginia and the Sunnyside bed in Utah, were selected for study and preliminary tests. Concurrently, the U.S. Geological Survey in Utah began a mapping and charting survey in an attempt to correlate occurrences of bumps with surface features.

A mining shield has been developed to protect miners and other underground workers at the working face from the hazards of falling rock and other materials. It can be manipulated to move (by telescopic motion to provide continuity of support) and follow the mining equipment. The shield will be produced commercially for the industrial market.

Coal Mine Injuries and Fatalities

According to preliminary reports received from coal-producing companies and State mine officials, the injury-frequency rate (number of injuries per million man-hours of exposure) was higher in 1957 than in the preceding 3 years. More than 47 men were injured (fatally or nonfatally) in accidents at bituminous coal, lignite, and anthracite mines in the United States in each million man-hours of time worked in 1957. There were 477 fatalities in 1957—the highest number of mine deaths in the 5-year period, 1953-1957—64 men were killed in five major disasters. Accidents involving falls of roof and face, the greatest hazard in underground coal mining[23 24/], caused 227 deaths, or 54 percent of the underground fatalities at all coal mines in 1957.

Reports on 51 fatal injuries at the anthracite mines of Pennsylvania in 1957 revealed that 49 were in deep mines, and 2 were on the surface.[25/] Falls of roof, face, or rib caused the deaths of 30 persons or 59 percent of all fatal accidents; underground haulage accidents were responsible for 5 fatalities, or 10 percent. Other fatalities were caused by gas explosion, explosives, falls, and other accidents. Twelve of these fatalities occurred in mines employing fewer than 15 men underground; 2 at an independent breaker not connected with an underground mine, and 37 (74 percent) at mines employing 15 or more men underground.

Accident Analysis Code

An accident-analysis code for roof-fall accidents was developed for establishing areas where new or additional research is necessary; this code points out critical areas that require protection, and permits the gathering and the publication of necessary facts for industry. The analysis sheet (to be

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23/ Bureau of Mines, Falls of Roof: The No. 1 Killer in Bituminous-Coal Mines, 1957: Mineral Industry Surveys HHS No. 462, 1958, 12 pp.

24/ Joseph, R. D., Causes of Roof-Fall Fatalities in Anthracite and Bituminous-Coal Mines, 1955 and 1956: Bureau of Mines Inf. Circ. 7869, 1958, 26 pp.

25/ Moore, James A., Fatalities at Pennsylvania Anthracite Mines, 1957: Bureau of Mines HHS No. 461, Mineral Industry Surveys, 1958, 20 pp.

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prepared by investigators) calls for information on 43 measurable dimensions, including support plans, location, and causes of accidents.

Safety Standards for Surface Auger Mining

The method of using large-diameter augers to recover coal was introduced to the mining industry about the end of World War II, and production of bituminous coal with augers has increased steadily to an estimated 7 million tons (approximately 1.4 percent of the total mined) in 1956. Although auger mining has proved to be reliable and economical, it has also been responsible for a number of fatal accidents caused by collapsing or sliding highwalls, asphyxiation, gas and/or dust ignitions, and other factors. Safety standards to prevent or combat the hazardous conditions associated with auger mining have been recommended.26/

Control of Fires in Coal Mines

Mine fires throughout the history of coal mining have resulted in high death tolls and considerable financial loss. These losses could have been reduced if adequate firefighting facilities had been available. Although it would be virtually impossible for every mine to maintain all of the facilities that are needed to extinguish widespread mine fires, practical facilities are available and their adoption would undoubtedly benefit the coal industry.27/

Uncontrolled fires in inactive coal deposits have posed similar problems. Since 1949 Congress has provided funds for the control of coal fires in inactive coal deposits. The Bureau extinguished or controlled a total of 61 such fires up to June 30, 1958. Thirty-five of these fires were on the public domain in the West, and the remainder on private property in the East. The Federal Government pays for fire control on the public domain, but the owner, State, or municipality must pay at least 50 percent of the cost incurred in controlling fires on private lands. An estimated 288 million tons of coal have been conserved under this program, at a cost to the Government of less than one cent per ton. Six fires were extinguished or controlled in 1958 (three on public domain) and attempts to control five additional fires were made at the end of the year. Maintenance work was also continued on several projects completed in earlier years, to control erosion of the cover banks and to re-cover spots where cracks appeared.

Mine Lighting and Mining Equipment

Continuing research to develop or improve mine-lighting systems and expanded use of approved systems should help to overcome one of the greatest handicaps to mine safety and production, that of poor visibility. Two additional fluorescent lighting systems designed by one company were approved

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26/ Bureau of Mines, Recommended Safety Standards for Surface Auger Mining: Bureau of Mines Inf. Circ. 7845, 1958, 11 pp.

27/ Van Natter, P. C., Fire-Protection System, Allen Coal Mine, Colorado Fuel and Iron Corporation, Stonewall (P. O. Weston), Las Animas County, Colo.: Bureau of Mines Inf. Circ. 7852, 1958, 23 pp.

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during the year, and new diaphragm-type connectors were certified for use in permissible alternating-current mine lighting systems. Preliminary work on the design of a small photoflash unit, weighing 4-1/2 pounds, was completed during the year. The housing of the unit is explosion-proof and the external circuit for triggering the flash device is intrinsically safe.

Considerable emphasis is being placed on the development of circuits that are intrinsically safe for mining machine controls. Operating designs are constantly being submitted by major companies to the Bureau for tests to determine the safety of the circuits in explosive natural gas-air mixtures, and many of the circuits had to be modified as a result of these safety studies. Remote control of mining machines used in gassy coal mines is of immediate interest and will have even greater importance in the future. Efforts are being made to develop methods for practicable operation of continuous mining machines by an operator stationed away from the machine. The Jeffrey MM76AM Colmol was redesigned to operate as a remote-controlled machine, the first of this type approved by the Bureau for operation in a coal mine. A Joy Manufacturing Co. boring type miner, with remote electronic controls, also has been studied.

As a result of the increasing use of roof bolting for roof support in coal mines, attention was directed to the need for control of the dust produced in the drilling process. Such dust is usually much higher in harmful free silica than dust produced by drilling or cutting the coal face. Since 1952 the Bureau has provided facilities for testing and approving drill-dust collectors for use with rock drilling in coal mines. Thirteen approvals were granted on newly designed drill dust collectors in 1958, bringing the total of approved collectors to 59. Numerous minor modifications in previously approved equipment were covered by extensions of approval. Recent designs tend toward the development of percussion and rotary drilling equipment in which dust and cuttings are removed from the hole, passing through hollow drill steel to a filter. These designs eliminate the necessity for keeping a hood or cup around the drill steel and against the roof to collect dust as it emerges from the hole.

Mine Ventilation

As part of the Bureau's program of coal-mine inspection under the provisions of the Federal Coal Mine Safety Act, approximately 18,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines, to detect and aid in the elimination of health and safety hazards from flammable and toxic gases in mines, and to provide information necessary in controlling and extinguishing fires in coal mines. Conventional gas-volumetric methods of analysis were supplemented by infrared spectrometry to determine, qualitatively and quantitatively, the methane content of certain coal-mine air samples of particular importance.28/ Other studies of important ventilation problems confronting the coal-mining industry were continued.

28/ Colbassani, P. J., and Watson, H. A., Determination of Low Concentrations of Methane in Coal-Mine-Air Samples by Infrared Absorption Spectrometry: Bureau of Mines Inf. Circ. 7839, 1958, 13 pp.

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Coal mining has been revolutionized by the continuous miner. This machine has replaced or combined into one step three formerly distinct operations: cutting, blasting, and loading. However, the multipurpose continuous miner introduced a new hazard—more rapid liberation of methane. A coal output formerly obtained from 10 to 20 working places can now be obtained from one. Although air circulation was generally increased, the bulk of this machine virtually precludes continuous flow of air across the face, where gas may accumulate.29/

Apprehension about this possible hazard led the Bureau of Mines to test face atmospheres during operation. Studies by the Bureau indicated that real danger may be present in many otherwise well-ventilated mines. Possible corrective measures are being explored. Research on face ventilation, where continuous-type mining machines are used, was conducted under controlled test conditions in the experimental mine at Bruceton, Pa. Field data also were collected in Pennsylvania and West Virginia mines to supplement the experimental results.30/ Field work has begun on the effectiveness of face ventilation when conventional coal-mining units are used.

Problems associated with the use of multiple main fans to ventilate bituminous coal mines were studied.31/ Suggestions were made for eliminating hazards due to faulty or improperly operated doors in the ventilating circuits of gassy mines.32/

Continuous Monitoring Methane System

A program was begun during the year to eliminate dependence on the human element for gas detection by development of a continuous methane-monitoring system and thereby reducing the number of mine disasters. The device is to be mounted on face-area equipment to give an alarm when the methane concentration reaches 1 to 1.5 percent, and to automatically stop machines when over 2 percent methane is detected. The program falls into two parts, development of (1) a detecting device capable of sampling the atmosphere continuously or at any interval desired, and (2) the necessary circuit to deenergize the trailing cable at its outby end. Several private instrument manufacturers are also engaged in this development program, and two of these companies have already demonstrated models of detecting devices to the Bureau.

The Bureau has conducted a comprehensive literature survey on known methods of detecting methane. Hot-wire oxidation of methane and modifications of this method that would increase the life span and ruggedness of the filaments

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29/ Stahl, R. W., Auxiliary Ventilation of Continuous Miner Places: Bureau of Mines Rept. of Investigations 5414, 1958, 16 pp.

30/ Kingery, D. S., Ventilation Problems in Connection With Continuous Mining Machines: Min. Cong. Jour., vol. 44, No. 9, September 1958, pp. 62-68.

31/ Kingery, D. S., Integrating Multiple Fans: Coal Age, vol. 63, August 1958, pp. 88-92.

32/ Kingery, D. S., and Harris, E. J., Coal-Mine Ventilation Without Doors to Control Main Air Currents: Bureau of Mines Inf. Circ. 7853, 1958, 13 pp.

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and reduce the temperature at which catalysts oxidize methane are being studied. A tentative list of specifications for methane monitoring systems was prepared as a guide for manufacturers working on these devices.

Safety Training and Education

First-aid training has been an important activity of the Bureau of Mines since its establishment. From 1910 to 1926 first aid was taught only by trained Bureau of Mines employees, but since that time, cooperative first-aid training programs have been practiced. Many mines and plants have taken advantage of this cooperative plan, whereby keymen in a mine or plant are given intensive training by a Bureau of Mines instructor, and subsequently serve as instructors to other personnel in their organization.

Since its organization in 1910 the Bureau of Mines had also assisted mining companies and other organizations connected with the mineral industries to conduct first-aid and mine rescue contests by providing instructors to train teams, judges, and other officials; by furnishing problems, and contributing in other ways, as requested.[33/] A safety course for bituminous coal-mine officials has been formulated, revised to incorporate the latest technical developments, and published for use by Bureau instructors, mining company officials, and other personnel who take the course.

Training in a new course, Fundamentals of Coal Mine Accident Prevention, was started in April 1958. This preliminary, nontechnical course was developed as a new approach to accident prevention, and should be followed by more technical courses.

STORAGE AND PREPARATION

Lignite represents a major national solid fuel reserve; extensive deposits occur in Texas, western North Dakota and eastern Montana, and smaller deposits in adjoining areas of Wyoming and South Dakota. The moisture content of lignite ranges from 35 to 40 percent and limits the consuming area area where this product can compete with higher-rank fuels. Most of the lignite produced, about 2-1/2 million tons in 1958, is consumed by power plants; smaller and decreasing amounts are used for domestic heating in North Dakota, Minnesota, and South Dakota.

Storage of Lignite

Lump size lignite for domestic uses is never stored for long periods because of degradation and dust formation, and minimum industrial stockpiles are maintained due to the possibility of spontaneous ignition.

Exposure of lignite to the atmosphere results in moisture release and, subsequently, in size degradation, dust formation, and susceptibility to

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33/ Weaver, H. F., and Alden, D. M., National First-Aid and Mine Rescue Contest, Louisville, Ky., October 2-4, 1957: Bureau of Mines Inf. Circ 7850, 1958, 68 pp.

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oxidation and spontaneous combustion. Stable, long-term storage of lignite has been demonstrated, however, at Garrison Dam, Riverdale, N. Dak., where more than 2 million tons of lignite have been successfully stored for 11 years. The method of stockpiling was developed by Bureau of Mines personnel in cooperation with personnel of the Army Corps of Engineers.

The piles are examined periodically and the heating value is determined annually. Comparisons on a moisture- and ash-free basis show an overall decrease of 1-1/2 percent in heating value from August 1953 to August 1958.

Pelletizing of Lignite

Pelletizing techniques were used in an investigation to produce a stable, storable fuel from small-particle lignite obtained as a waste material during preparation of commercial sizes.34/

A North Dakota Lignite was processed in a rotary-drum retort at temperatures from 250° to 950° F., and the quantity, composition, and heating value of the degradation products were determined to establish optimum conditions for pelletizing. Heat balances indicated that essentially 100 percent of the potential heat was recovered in the dry solid residue produced at temperatures below 500° F., but was only 81.3 percent at 950° F. because of increased formation of gas and tar. The influence of process variables on the properties of the pelletized material was investigated in a pelletizing drum with various sized feeds, starch or bituminous binders and different mixing and retention times. Both starch-base and bituminous-base pellets were free-flowing and dust-free and would not slack on storage. Bituminous binder was generally superior to starch binder.

Freezeproofing of Lignite

Vaporization of the moisture in lignite during winter transport, and recrystallization as frost on the coal particles adjacent to the car wall tend to cement particles together and produce agglomeration. The factors which contribute to this agglomeration by freezing and the development of improved methods of eliminating or alleviating the problem are being investigated.

A method for determining the degree of agglomeration of laboratory samples was developed to a point where satisfactory reproducibility was obtained.

Various treatments and additives have been compared. Oil treatment of lignite at the rate of 3 to 5 quarts per ton has been found partly effective in reducing the degree of agglomeration. None of the additives tested, including commercial spray oils, fuel oils, industrial solvents, a water-soluble oil, lubricating oils, rock salt, calcium chloride, ethylene glycol, methyl and ethyl alcohol, was superior to, or as effective, as the commercial spray oils

34/ Oppelt, W. H., Cooney, J. P., Golob, E. F., and Kube, W. R., Thermal Pretreatment and Pelletizing of North Dakota Lignite: Bureau of Mines Rept. of Investigations 5382, 1958, 55 pp.

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now used. However, the most promising method of reducing agglomeration that has been tested appears to be the admixing of dried lignite to the as-mined lignite. A pronounced reduction in degree of agglomeration was found when 10 percent by weight of partly dried lignite (25 percent moisture content) was mixed with a sample of normal lignite. Agglomeration was reduced even further when 10 percent of dried lignite (5 percent moisture content) was added. Agglomeration was eliminated in samples containing 20 percent by weight of the dried lignite. The mixing of dried lignite as a freezeproofing agent offers advantages not only from the standpoint of reducing the freezing characteristics of this material, but also in terms of the increased heating value that can be effected. These and other factors, for example, content of dried lignite, mixture ratios, and effect of size consist on degree of agglomeration, are being further evaluated.

Hydraulic Transportation of Lignite

In 1958 a report was published[35/] describing the Bureau's work on the hydraulic transportation of a Texas lignite. This work was conducted in 1955, and also included some studies on a Colorado high-volatile bituminous coal.

Pipeline tests simulating 75 miles of movement caused extreme degradation with the lignite, severely aggravating the dewatering and drying problem at the discharge end of the line. Estimated total cost for transport, dewatering, and drying lignite to zero surface moisture, extrapolated to 5,000 tons per stream-day over a distance of 100 miles, is 3.0 cents per ton-mile. Under similar test conditions, degradation of bituminous coal was much less than of lignite. As a consequence, dewatering and drying cost estimates are lower.

Size Reduction of Lignite

In its natural state, lignite is tough, resilient, and resistant to fracture. With the use of pulverized-fuel firing increasing, information on the pulverization characteristics of lignite would be helpful.

Study of the size reduction of lignite in an experimental impact pulverizer was therefore continued. Tests indicate that power requirements decrease with decreasing moisture content. Stable and controlled pulverization of predried samples was achieved with room air circulating through the mill. Use of natural lignite with inert gas preheated to controlled temperatures for sweeping the test mill is being studied.

Mixing and Segregation

Because mixing and segregation are of the greatest importance in all phases of coal preparation and utilization, a comprehensive investigation of this problem is being conducted. Present efforts are directed toward small-scale laboratory studies of model systems, and are intended to provide information about the mechanism of mixing of particulate solids.

35/ Lammers, G. C., Allen, R. R., Donaven, D. J., Wagner, E. O., and Parry, V. F., A Study of the Feasibility of Hydraulic Transport of a Texas Lignite: Bureau of Mines Rept. of Investigations 5404, 1958, 39 pp.

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During the year, a new mathematical criterion was developed to denote the extent of mixing for multicomponent mixtures.36/ Preliminary investigations indicated that this criterion provides a convenient measure of the degree of mixing, and is well suited to the determination of mixing rates for a wide variety of systems, including many of industrial importance. The mechanism of mixing, the effects of mixer design and operating variables on mixing rates are being studied. The fundamental information derived from these studies should be helpful in the subsequent study and evaluation of large-scale equipment and the mixing of heterogeneous solids such as coal and other minerals.

Mechanism of Flotation

One of the most difficult problems in coal preparation is the cleaning and dewatering of the fine sizes. Since this material is not responsive to cleaning methods based upon differences in the specific gravity of the coal and its associated impurities, cleaning processes based on differences in wetting characteristics seem to be most promising for treating the fine sizes. Therefore, the theory and practice of coal flotation are being studied and the results should contribute to the development of more efficient preparation methods and more complete utilization of our coal reserves.

Emphasis was continued on elucidating the mechanism of froth and kerosene flotation and determining the influence of operating variables on cell capacity. Results confirmed earlier findings that aeration is one of the more important variables in influencing flotation rates. To permit detailed studies of this factor, an improved method was devised for measuring the rate of aeration at the surface of an operating cell.37/

A study of the effects of coal oxidation on flotation rates was initiated. Coals were oxidized for different lengths of time in an autoclave at 100° C., and the floatability of these coals was then determined in batch- and continuous-type flotation cells. Initial results indicate that the type of flotation reagent used has a marked influence on the floatability of oxidized coals.

Because the separation of coal from refuse by flotation-type processes depends on the ability of the coal particles to come into contact with, and adhere to, air bubbles, contact angles and the factors influencing them are being studied. The effects of oxidation and temperature were evaluated, and contact angles were measured for a number of coals and related substances.

Fine-Coal Cleaning

The Bureau is interested in improved methods for cleaning fine coal as a means of minimizing waste in preparation, making substandard coals available

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36/ Gayle, J. B., Lacey, O. L., and Gary, J. H.. Mixing of Solids- Chi Square as a Criterion: Ind. Eng. Chem., vol. 50, No. 9, September 1958, pp. 1279-1282.

37/ Gayle, J. B., Improved Method for Measuring Aeration in Flotation Cells: Min. Eng., vol. 10, No. 7, July 1958, p. 796.

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for use, and providing industry with fuels of better quality at lower cost. Therefore, a program is being conducted to investigate promising coal-preparation processes.

One of the methods investigated is the feldspar jig which is used widely in Europe to clean fine coal. Preliminary tests were made with a laboratory unit, and were followed by tests of the first full-size jig installed in the United States.38/ Three coals having distinctly different washability characteristics were used in the plant tests, and the jig was used to treat both sized (1/4-inch to 48-mesh) and unsized (1/4-inch to 0) feeds. The full-size jig had a capacity exceeding 1 ton per hour per square foot, which is much greater than that of fine-coal cleaners generally used in this country; such high capacity offers distinct economy in plant construction. Effective cleaning down to 48 mesh was accomplished but, as is the case with other fine-coal cleaning devices, finer impurities were not removed completely.

The dense-medium cyclone is another fine-coal cleaning process being investigated. A number of cyclone plants have been installed in Europe in the last few years,39/ and an inspection of these plants disclosed that the typical cyclone-plant flowsheet does not differ greatly from that of conventional coarse-coal, dense-medium plants in this country, except for the use of the cyclone as the separating vessel.

In contrast to European practice where the extreme fines are removed from the feed to the cyclone washer and treated separately by froth flotation, the Bureau is attempting to wash 1/4-inch by 0 coal. A series of twenty tests were made to study the effects of varying the specific gravity of the dense medium, feed inlet pressure, coal solids concentration and the diameters of the overflow and the underflow orifices. After each test the clean coal and refuse products were screened at 14-mesh and the 14-mesh by 0 fractions separated into magnetics and nonmagnetics. The 1/4-inch by 14-mesh products and the nonmagnetic fraction of of the 14-mesh by 0 products were weighed and analyzed for ash. Effective cleaning with high recovery efficiency was achieved, especially in the 1/4-inch by 14-mesh size fraction. Recovery of magnetite from the 14-mesh by 0 products was incomplete, and analyses of these fractions showed inordinately high ash contents.

The problem of cleaning fine coal in dense medium, either in cyclones or static baths, is inextricably linked with the characteristics of the medium and their influence on the movement of coal and other particles in the cleaning vessel. The effect of medium density, viscosity, and yield value on particle movement are being investigated. Laboratory tests indicate that some clays, particularly bentonites, greatly increase the yield value of a magnetite medium; however, in such medium small coal particles having a density close to that of the medium are unable to initiate movement from a position of rest.

38/ Geer, M. R., and Yancey, H. F., Operating Results With the Feldspar Fine-Coal Jig: Bureau of Mines Rept. of Investigations 5412, 1958, 14 pp.

39/ Yancey, H. F., Cyclone Washers for Fine Coal: Coal Age, vol. 63, No. 12, December 1958, pp. 118-121.

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The data on particle movement suggests that the yield value of a medium is more important than viscosity in affecting cleaning results.

Evaluation of processes for cleaning fine coal, or for other coal cleaning processes, depends on float-and-sink tests, but no recognized standard method is available. A technique has been recently developed for float-and-sink testing fine coal in a centrifuge. Data from 220 tests indicate that minus-14-mesh coal can be separated in about 20 minutes at a centrifuge speed of 1,000 r.p.m. (230 gravs.) with an accuracy of ± 2 percent. The results compare favorably with static methods which require 24 or more hours in a constant temperature bath for complete separation.

Dewatering Coal Slurry

The recovery of fine coal from washery water is becoming increasingly important in efforts to improve coal recovery and to permit reuse or discharge of water as waste. Techniques for liquid-solid separation are being studied and developed. An investigation of flocculation as an aid to vacuum filtration has been extended to include additional slurries of different composition. Evaluation of the DSM (Dutch State Mines) Sieve Bend was continued,[40] and high capacity and efficiency were obtained in laboratory tests with a commercial-size unit. Screening efficiencies up to 78 percent could be obtained when making a screening separation as fine as 48 mesh; peak efficiency was obtained at flow rates approximating 300 gallons per minute of slurry per foot of screen width.

The cost of removing water from fine coal by thermal drying is very high. Increasing the moisture-removing capacity of a vacuum filter cake during the drying phase of the filter cycle could reduce costs.

The feasibility of reducing filter cake moisture by applying gas-fired radiant heat to the filter cake is being studied.

Preliminary tests with minus 48-mesh coal slurries indicate that the cost per ton of water removed with this technique is about half that of thermal drying.

Preparation Characteristics of Coking Coal

The survey of washability characteristics of coals on a county basis, begun in 1948, was continued. Bureau reports for two Virginia counties were published during 1958. Similar studies were made on beds in other states. Figure 5 shows the counties for which reports are published and the status of uncompleted reports.

40/ Geer, M. R., and Corp, L., Test Performance of the Sieve Bend: Mechanization, vol. 22, No. 4, April 1958, pp. 104-105.

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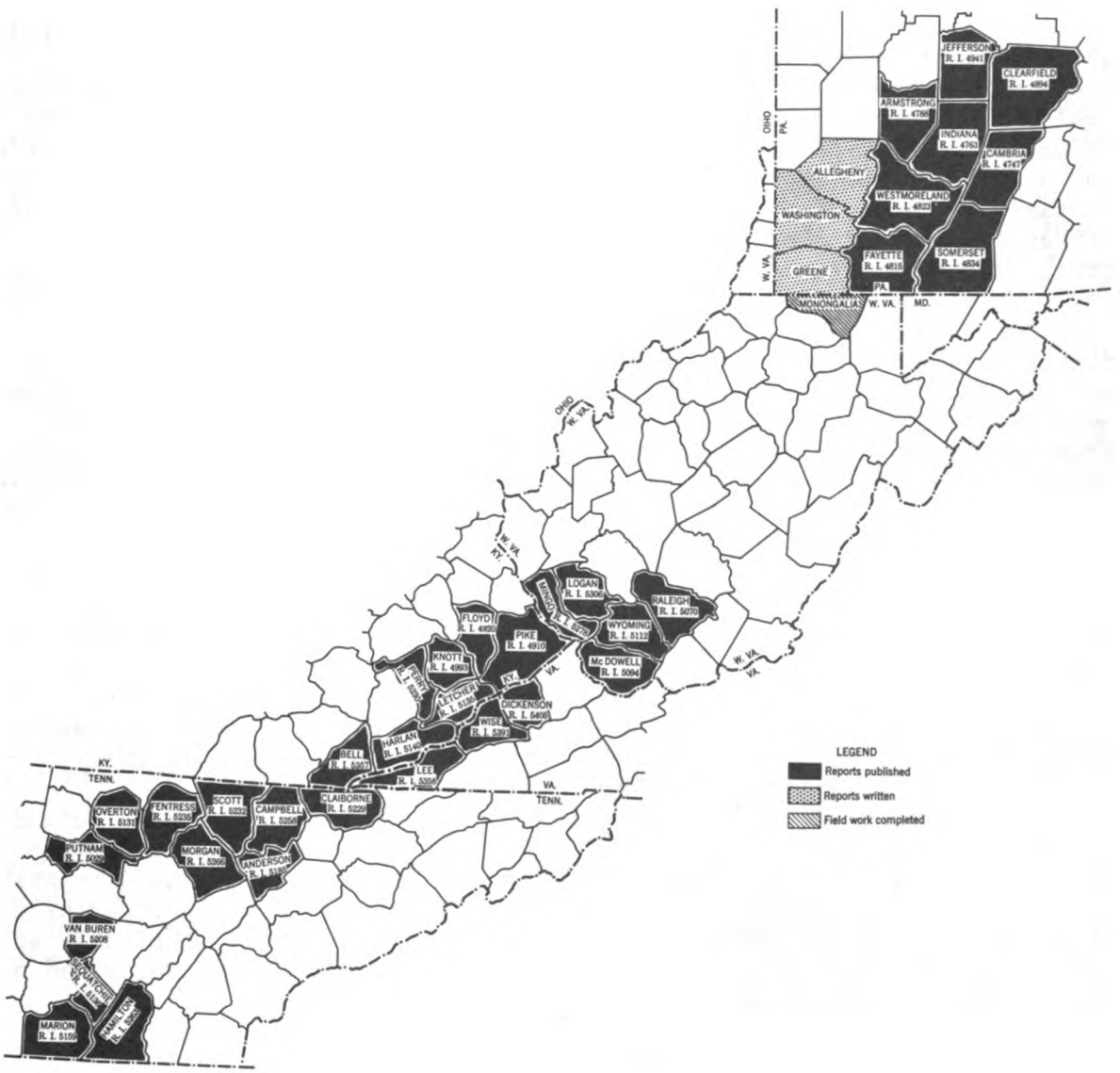

FIGURE 5. - Map Showing Status of Washability Reports on Coking Coal, 1958.

Wise County, Va.

Wise County is the second most important coal-producing county in Virginia; approximately 6-1/2 million tons of coal were produced in 1955, and about one-third of this output was cleaned in six mechanical cleaning plants.[41]

41/ Gray, T. E., and Boley, C. C., Preparation Characteristics of Coal From Wise County, Va.: Bureau of Mines Rept. of Investigations 5391, 1958, 50 pp.

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The Taggart and Clintwood beds are important from both the standpoint of production and estimated reserves. The Taggart bed is chemically suited for making metallurgical coke without mechanical cleaning, while the Clintwood coal can be cleaned by a simple washing process. The Dorchester bed, which has the largest estimated reserves and small production, varies so widely in sulfur content that in one area no mechanical cleaning is necessary to prepare a coal satisfactory for making a metallurgical fuel. In another area, the coal presents a simple cleaning problem, while in a third area the coal is too high in sulfur to be upgraded to metallurgical standards by usual commercial processes. Other beds are of minor or no importance from the standpoint of reserves and present production. No cleaning is necessary for the High Splint, Taggart Marker, Blair, and Lyons beds. The Kelly and Imboden beds are chemically suitable for metallurgical use without mechanical cleaning, except that the sulfur content in one location of each bed cannot be reduced enough by ordinary commercial processes so the product can qualify as a metallurgical fuel. The Norton, Upper Banner, Lower Banner, Kennedy, and Raven beds present simple cleaning problems. The Jawbone bed may be cleaned to a chemically suited metallurgical coal if the ash specification is not too rigid. Finer crushing before washing has little effect on the liberation of impurities.

Dickenson County, Va.

Dickenson County is the third most important coal-producing county in Virginia;[42] more than 3-1/2 million tons of coal were produced in 1955, of which approximately 70 percent was mechanically cleaned in five cleaning plants. Almost 60 percent of the coal mined was from the Clintwood bed. Among other beds sampled, the Upper and Lower Banner, Kennedy, Raven, and Jawbone beds are the most important from the standpoint of estimated reserves.

The coal reserves remaining in the Clintwood bed are chemically suited for metallurgical use as mined. Coal from the Eagle, Norton, Splash Dam, Upper Banner, Lower Banner, Jawbone, and Tiller beds can be upgraded by washing to be chemically suitable for metallurgical purposes. The Dorchester coal must be washed at a low specific gravity to produce a product chemically suitable for metallurgical use. Generally, finer crushing before washing had no effect on the release of impurities.

Greene County, Pa.

Work on the washability of coals in Greene County has been completed.[43] The results show that the Pittsburgh coal at six locations may be used for metallurgical coke after washing, and that at two locations this coal has a sulfur content which is still too high after washing to be used unblended as a metallurgical fuel. The Sewickley and Waynesburg coals are not suitable for metallurgical fuel under present conditions. Finer crushing for the liberation

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42/ Gray, T. E., and Boley, C. C., Preparation Characteristics of Coal From Dickenson County, Va.: Bureau of Mines Rept. of Investigations 5405, 1958, 31 pp.

43/ Gray, T. E., and Palowitch, E. R., Preparation Characteristics of Coal from Greene County, Pa.: Bureau of Mines Rept. of Investigations 5434, 1958, 31 pp.

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of impurities before washing had little effect on the Waynesburg and Sewickley coals, but had a somewhat greater effect on the Pittsburgh coal.

Washington County, Pa.

Results of the completed study of Washington County show that the Pittsburgh coal in four locations is suitable for making metallurgical coke if washed at a low specific gravity, and when necessary, crushed to a finer size before washing; at two locations this coal contains too much ash and sulfur after washing for metallurgical use. The Redstone coal may be upgraded to a metallurgical coke if cleaned at a low specific gravity or crushed to a smaller size before washing at a high specific gravity. Finer crushing for the release of impurities before washing had an effect varying from slight to appreciable on the Pittsburgh coal, but had a substantial effect on the Redstone coal.

Allegheny County, Pa.

Results of the washability studies in Allegheny County indicate that the Pittsburgh bed is suitable for metallurgical coal if washed at a low specific gravity. The Redstone-bed coal can be upgraded to a chemically suited metallurgical coal by mechanical cleaning. All but one sample of the Upper Freeport coal showed that it can be upgraded to a metallurgical fuel by mechanical cleaning at suitable specific gravities. Finer crushing of these coals before washing showed some release of impurities.

Pierce County, Wash.

In addition to the regular coking-coal survey, the washability characteristics of deposits in Pierce County are being examined. The coals in this county constitute the only reserve of coking coal on the Pacific coast. They have high coking strength and low sulfur contents, but some of them are unusually difficult to clean to the required ash content. The Bureau, in cooperation with a private company that is exploring the field, is evaluating various cleaning methods applicable to sizes too fine for conventional dense-medium treatment. This evaluation is made in an effort to develop a suitable flow sheet which would permit even the most difficult of these coals to be utilized.

Mary Lee and Pratt Beds, Ala.

In 1955 the Bessie washer of the U.S. Pipe and Foundry Company was destroyed by fire and company officials requested the Bureau to cooperate in a washability study of the Mary Lee bed near Maben, Ala., to obtain sufficient data to permit selection of the equipment needed for an efficient cleaning plant. Based on the results of this study[44] and experience from previous installations, a new plant was constructed and is currently producing a coking coal with ash and sulfur contents averaging 8.8 and 0.85 percent, respectively, without the production of steam coal.

44/ Riley, H. L., Perry, R. E., and Gayle, J. B., Washability Study of Coal From Mary Lee Bed, Maben, Ala.: Bureau of Mines Rept. of Investigations 5393, March 1958, 14 pp.

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In further cooperation with this company, the preparation characteristics of both the upper and lower benches of the Pratt bed near Graysville, Ala., were investigated.45/ Preliminary results indicated that reducing the sulfur content to an acceptable level constitutes the major problem in cleaning this particular coal. Laboratory washability tests indicated that separation at an unusually low gravity might yield an acceptable product if such a separation were technically and economically feasible. Accordingly, a plant test was carried out in which 500 tons of coal were processed in the new Bessie washer. For this test the heavy-media unit was adjusted for a gravity separation of 1.30 and the table and the flotation units adjusted to give as nearly comparable separations as possible. The ash content of the cleaned coal was less than 5 percent, but the sulfur content (1.7 to 1.8 percent) precludes its use except in relatively small amounts in coking blends under present specifications for metallurgical cokes.

Washability Study of Henryetta Coal, Okmulgee County, Okla.

A sample of Henryetta coal from the Blackstone mine, Henryetta, Okla. was crushed in stages to sizes passing through 1-1/2-inch, 3/8-inch and 14-mesh and each of the resulting size fractions were float-and-sink tested. The data indicate that washing the 1-1/2-inch by 100-mesh at a low specific gravity (1.30) theoretically yields 79.9 percent float coal containing 2.2 percent ash and 0.98 percent sulfur; washing the 1-1/2-inch by 100-mesh at a high specific gravity (1.60) theoretically yields 93.8 percent of float coal with 3.3 percent ash and 1.14 percent sulfur. Crushing the sample through 3.8-inch and 14-mesh prior to separation indicated some increase in recovery, slight reduction in float ash content, but no reduction in float sulfur content.

Washability Study of Lower Freeport Coal, Somerset County, Pa.

A sample of the Lower Freeport bed coal from Somerset Co., Pa. was crushed to a top size of 1-1/2-inch, and screened at 4- and 14-mesh. The resulting size fractions were float-and-sink tested. Market requirements for this coal demand a maximum ash content of 7.5 percent. The tests made indicate that coal of this quality can be obtained by several alternative procedures. To prepare a coal with 7.5 percent ash the minus-4-mesh fraction could be washed at about 1.60 specific gravity and the 1-1/2-inch by 4-mesh fraction at approximately 1.38 specific gravity, which would result in an overall theoretical recovery of 87.5 percent of the 7.5-percent-ash coal. Approximately 96 percent of the plus-4-mesh size material is in the near gravity range (± 0.10) at 1.38 specific gravity. The 1-1/2-inch by 4-mesh fraction therefore could be washed in a dense-medium washer at 1.34 specific gravity which would theoretically produce a 6.1 percent ash coal at a yield of 63.3 percent of the feed and blended with the unwashed 4-mesh by 0 fraction, resulting in an overall ash content and yield of 7.4 and 78.3 percent respectively. This scheme would eliminate the fine coal cleaning problems at a slightly reduced overall

45/ Perry, R. E., Gandrud, B. W., Riley, H. L., Gayle, J. B., and Eddy, W. H., Laboratory and Full-Scale Sulfur Elimination Tests on Coal From Pratt Bed, Alabama: Bureau of Mines Rept. of Investigations 5397, May 1958, 25 pp.

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recovery. Alternatively, the run-of-mine 1-1/2-inch by 0 material could be washed in a jig, or the plus 4-mesh by dense medium and the minus-4-mesh by air tables.

Preparation of Low-Ash Coal for Carbon Electrodes

A 1-ton batch flotation test was made using Eagle-bed coal from Wyoming County, W. Va., ground through 20-mesh. By successfully recleaning the concentrate seven times it was possible to reduce the ash content of the raw coal from 5.8 to 1.4 percent with a recovery efficiency of 88.5 percent. Sodium chloride at 0.8 percent concentration was used as the flotation reagent.

Anthracite Preparation

The laboratory facilities for cleaning and sizing coal at the Anthracite Experiment Station have been expanded, and equipment is now available for complete washability studies, experimentation on heavy-media preparation of anthracite, the study of cyclones as cleaning and sizing agents, and for study of froth flotation as a fine-coal cleaning process (Figure 6). In addition, an air-swept ball-mill pilot plant has been installed for a study of fine-grinding of anthracite, and tests have been conducted to determine the effect of size on ash content of various-sized fractions of anthracite.

The work to date indicates that in order to apply heavy-media preparation techniques to fine sizes of anthracite, it is necessary to achieve very precise density control, and equipment has been obtained to attempt to supply the type of control necessary. Washability tests have been conducted on both Holmes-seam and Mammoth-seam anthracites, and the data are now being assembled in the form of a report of investigations.

COMBUSTION, BOILER WATER TREATMENT, AND POLLUTION

Heat Transfer in Furnaces

The efficient use of coal as a source of heat and power is of major importance in our economy. Attempts are being made to increase the efficiency of coal consumption in a continuing program which includes: The evaluation and correlation of the performance of various industrial and electric utility furnaces[46]; a study of reactions between ash and other combustion products responsible for ash and slag deposits; and a study of the combustion of pulverized coal in suspension.

Attention has been directed to the calculation of flame temperatures[47], and other factors involved in furnace efficiency. Total heat transfer in

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46/ Burdick, L. R., and Corey, R. C., Effect of Fuel Properties on Firing Methods: Proceedings of the Joint Conference on Combustion (The Institution of Mechanical Engineers, London, and The American Society of Mechanical Engineers), 1955, pp. 68-71.

47/ Myers, J. W., Goldberg, S. A., and Smith, R. W., Jr., Calculation of Theoretical Flame Temperatures in Furnaces: Trans. ASME, vol. 80, No. 1, January 1958, pp. 202-216.

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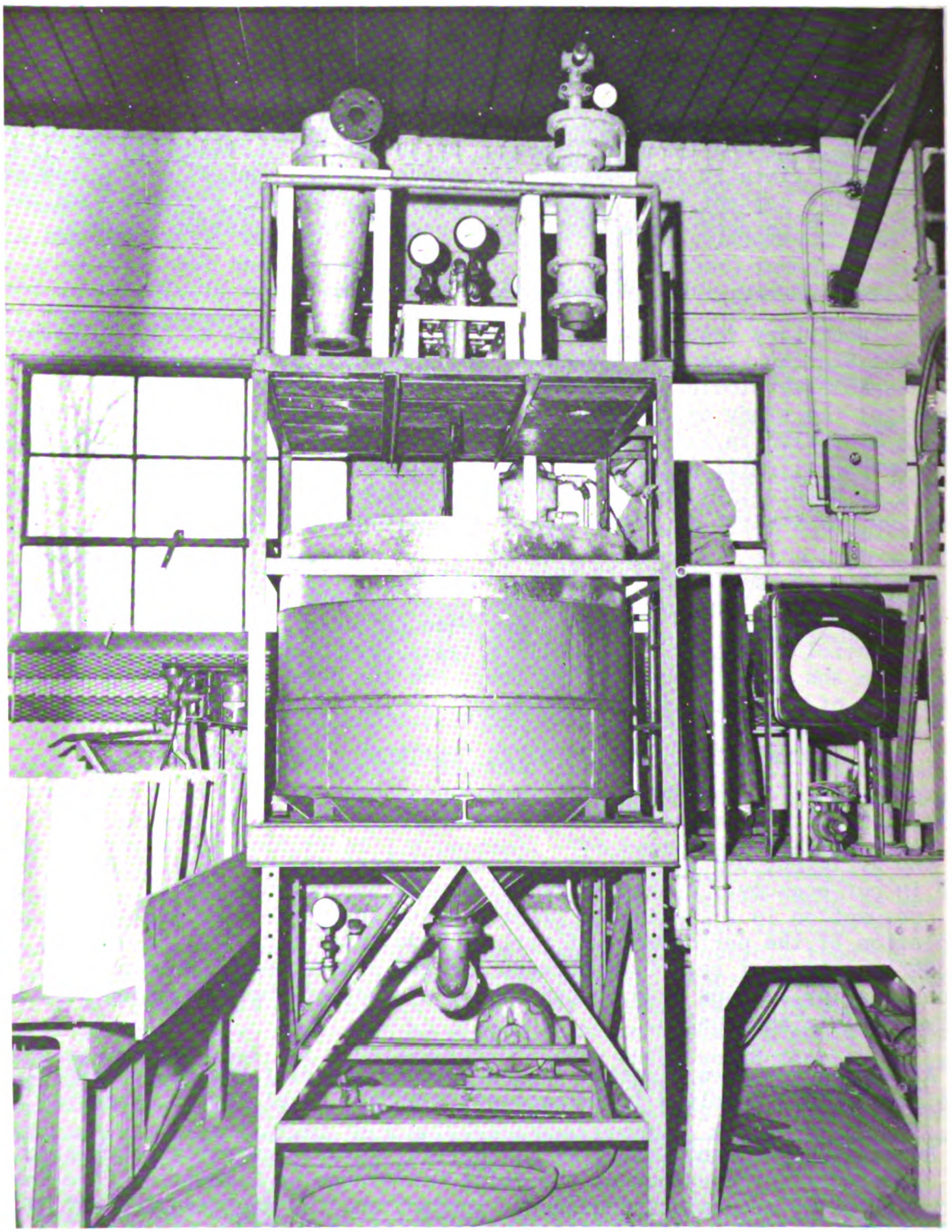

FIGURE 6. - Cyclone System in Coal Preparation Laboratory, Anthracite Experiment Station.

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boiler furnaces was studied in cooperation with the Special Research Committee on Furnace Performance Factors[48]. The correlation of the heat-transfer rates observed in these studies was suspended until instrumentation is developed to measure flame size and shape and the reflection of heat by the water-cooled furnace walls.

Ash and slag deposits on heat-transfer surfaces are responsible for reduced heat-transfer rates, obstruction of gas flow, and corrosion of metal surfaces. Troublesome deposits containing ash components that are quite different from the original coal ash are being studied. High-temperature reactions between other combustion products and coal ash are being investigated to determine the conditions under which the changes in composition and formation of deposits occur. Fly ash catalyzes the oxidation of sulfur dioxide to trioxide in flue gases and, at high temperatures, may gain or lose weight by reaction with the sulfur oxides. The direction and quantity of weight change is a function of several variables. Maximum gain in weight for two ashes which were investigated occurred at about 1200°F. Changes in the physical appearance and composition of fly ash occur when the material is subjected to high temperatures and high temperature gradients. These changes are being studied in laboratory tests. Selective concentration of sulfur, similar to that noted in deposits in industrial furnaces, has been obtained in the laboratory. However, migration of other ash components to the extent found in bonded deposits has not been observed.

Combustion of Coal in Suspension

A large part of the coal used for producing electric power is burned as pulverized coal in suspension. The conditions needed for complete burning in cold-wall furnaces have been established empirically. Flame velocities and other conditions needed for flame propagation are known for small flames burning from a few milligrams per minute to a few grams per hour. Studies directed towards extending such measurement to flames burning up to 5 pounds of coal per hour were begun. An apparatus for feeding coal in air at desired rates has been developed, and a furnace is being designed and constructed for the tests.

Fluid Mechanics

The dynamics of flowing fluids[49] is of importance in many phases of coal utilization. Analysis of data on the relationship between flow patterns, turbulence, heat transfer, and rates of combustion reactions was continued, with a view towards contributing to the solution of problems encountered in pneumatic transport of pulverized coal from storage to furnace, hydraulic transport of coal between mine face and washery or to an ultimate consumer, and instrumentation for furnace and air pollution measurements.

48/ Orning, A. A., Weintraub, M., Schwartz, C. H., Mihok, E. A., McCann, C. R., and Harrold, W. C., An Investigation of the Variation in Heat Absorption in a Pulverized-Coal-Fired Slag-Tap Steam Boiler at Blaine Island, Charleston, W. Va.: Trans. ASME, vol. 80, No. 6, August 1958, pp. 1239-1250.

49/ Weintraub, Murray, Flow of Fluids: Ind. Eng. Chem., vol. 50, No. 3, Part II, March 1958, pp. 447-452.

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Combustion of Lignite and Anthracite

Standard ASME tests were conducted on four different small industrial anthracite stokers, and a normal-use test of about 3-months' duration was conducted on one of these units. The efficiencies of the small industrial stokers (100-400 lb. per hour), equipped with reciprocating water-cooled grates, increased from 70 to 85 percent under continuous stoker operation at rated capacities from 25 to 100 percent. Complete data on these tests are being analyzed for publication.

Although the use of lignite for domestic heating has continuously declined over the years, this fuel can perform satisfactorily and economically in properly designed equipment. The performance of three domestic heaters (a hand-filled space heater, a magazine-type heater, and a ram-type automatic overfeed stoker) was investigated some years ago. Results from these tests were evaluated and analyzed during 1958. Lignite was readily burned in each of the three units at efficiencies up to 75 percent on a gross heating value basis. However, these efficiencies were obtained by close supervision of the furnaces and would probably be lower under normal household operating conditions.

Fuel-Engineering Service

Engineering assistance and technical advice were provided to other Federal agencies in the proper selection of fuels and in testing operations to determine the capabilities of steam generating and auxiliary equipment. At the request of other agencies, the following services were rendered:

General performance and efficiency tests were conducted on one of three new 150,000 pounds per hour combination oil- and pulverized coal-fired steam generators installed in the Goddard Generating Stations, U.S. Naval Powder Factory, Indian Head, Md. Furnace exit and final steam temperatures and thermal efficiency did not meet specifications. The manufacturers have proposed corrective measures to permit continuous operation in conformance with specifications.

Efficiency tests were conducted on a 20,000 pounds per hour natural gas- and oil-fired boiler installed in the Jim Thorpe Heating Plant, Haskell Institute, Lawrence, Kans. The calculated results of the tests, which were conducted on individual fuels, indicated that the boiler and its auxiliary equipment were capable of meeting specifications.

Performance and efficiency tests were conducted on a prototype vibrating stoker-fired 12,500 pounds per hour package steam-generating unit located in the Central Heating Plant, Kingsley Air Force Base, Klamath Falls, Oreg. The generating unit and its auxiliaries met specifications with the exception of the dust collector.

Visual observations were made during performance tests on a prototype oil- and vibrating stoker-fired 30,000 pounds per hour package steam generator in the Hospital Heating Plant, Great Lakes Naval Training Center, Great Lakes, Ill. Observations were made during tests on No. 6 fuel oil and on Indiana high-volatile bituminous coal. Observations will also be made on high- and low-volatile bituminous coals and lignite.

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Fuel-burning observations at Andrews Air Force Base and at the Pentagon led to recommendations on the proper selection of fuels.

Assistance was given the Department of the Army in preparing specifications for purchasing solid fuels for the Third, Fourth, and Sixth U.S. Armies, the Military District of Washington, the Chemical and Transportation Corps, the Adjutant General, and Engineer installations.

Personnel at Fort Jay, Governors Island, N. Y., were advised of corrective procedures for controlling flashback problems in connection with oil-fired heating boilers.

Preparations were made for future general performance and efficiency tests on two new boilers installed in the Central Heating Plant, Tuba City Subagency of the Navajo Indian Reservation, Tuba City, Ariz.

Boiler-Water Treatment

The boiler-water research program is essentially a fuel-economy project conducted in connection with the boiler-feedwater service supplied to Federal heating and power plants. Corrosion in steam-condensate return lines results in wasted condensate and is an important contributing factor to the inefficient use of fuel. Methods of feedwater treatment are available to bring corrosion problems under control once the nature of each problem and its severity have been determined. The tester developed by the Bureau permits a Federal plant to evaluate its return-line corrosion problem and determine the effectiveness of the preventive chemical treatment used.50/ A total of 344 such testers were evaluated in 1958 and 424 new tests were begun.

The most frequent cause of return-line corrosion is carbonic acid in the condensate, stemming from the alkalinity (bicarbonate content) of the makeup water. Problems resulting from the use of soda ash (sodium carbonate) in feedwater treatment can be corrected by substituting sodium hydroxide. Improved deaerating-heater operation minimizes recycling of carbon dioxide dissolved in the return condensate. As an alternative to neutralizing carbonic acid in steam condensate, many industrial plants use octadecylamine to form a continuous water-repellent coating separating and protecting the pipewall from the corrosive condensate. An investigation was made to determine the most efficient method of using filming amines, including a study of the concentration required and point of introduction. The problem is complicated because octadecylamine is unstable in a heating system, the principal degradation products being dioctadecylamine and ammonia. Analytical methods were developed for all three substances and improved until laboratory testing showed them to be adequate for plant survey work.51/

50/ Berk, A. A., Use of a Condensate-Corrosion Tester for the Survey of Return-line Deterioration: Corrosion, vol. 14, No. 3, March 1958, pp. 41-44

51/ Hopps, G. L., Getz, M. E., and Berk, A. A., Trace Concentrations of Octadecylamine and Some of Its Degradation Products: ASME Preprint 58-A-264, 1958, 8 pp.

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Surveys in about a dozen Federal and industrial plants confirmed the essential instability of octadecylamine in the hot steam and condensate. The liberated ammonia corresponded, within analytical accuracy, to the degree of degradation determined from the quantities of primary (octadecylamine) and secondary (dioctadecylamine) amines recovered. Degradation was usually about 70 percent complete at the trap for draining the condensate.

As degradation at the steam-trap discharge was rarely more than 75 percent of dosage, the steam condensing equipment, the traps, and some of the piping downstream from the traps may have been filmed adequately. Much of the iron in rust-colored condensate stems from these especially vulnerable surfaces so that even this limited control over corrosion may be very important. Essentially complete control could be obtained in industrial systems where comparatively little condensate piping was found beyond the traps; relatively heavy accumulations of greasy iron oxide were also found in Federal systems, which probably is due to the summer inactivity of heating systems.

The degradation of octadecylamine would be of little importance if the principal degradation product were an equally effective film-forming material. Laboratory testing under a variety of conditions has, however, failed to yield corrosion-preventing films from dioctadecylamine-treated condensate. Although the films formed from octadecylamine-treated condensate are actually dioctadecylamine, protection is temporary because these films are rapidly removed by hot condensate, and continuous treatment is necessary to maintain freedom from corrosion since the secondary amine is more readily dispersed by water. Laboratory testing has also shown that the degradation of octadecylamine does not stop with the formation of dioctadecylamine, but continues until all the nitrogen is released as ammonia, leaving the free hydrocarbon.

Because octadecylamine treatment has limited value for controlling corrosion in Federal heating plants, attention has been directed toward developing a more stable substitute.

Engineering Service

Boiler-water service is furnished to Government-operated heating and power plants on a continuing basis. Recommendations for increasing fuel efficiency and plant maintenance are based on the analysis of water samples and deposits, and information supplied by the plants regarding their equipment and operating procedure. Water samples are periodically analyzed to check the accuracy of control testing at the plant and to determine the efficiency of the boiler-feedwater treatment prescribed for that plant.

During the year more than 13,500 boiler water and other samples were analyzed for 16 Government agencies, and based on these analyses, recommendations on boiler-water treatment were made. Reports and recommendations were also made covering analyses of 10 boiler-plant scales and deposits, 387 miscellaneous special water samples, and a boiler-water treatment compound. More than 3,000 orders from Government agencies for boiler-water testing equipment were filled.

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Consulting service was provided to many agencies on boiler-feedwater conditioning and control testing, prevention of corrosion, and a variety of related problems.

The purposes and methods of operation of the boiler-feedwater service were described at the Navy Sanitary Engineering Conference for Field Personnel at Cincinnati, Ohio. Guidance was also given on some special boiler-feedwater problems.

The total number of plants being serviced increased about 1 percent in 1958, bringing the total number of boilers serviced to an estimated 9,900. Through the operation of this program, substantial savings are being realized by Government agencies in increased boiler efficiency and lower costs of plant maintenance and repair. Savings of $3,400 per boiler per year in fuel, maintenance, and replacement costs are reported by representative plants.

Air Pollution

Combustion Characteristics of Refuse

Cooperative work with the Public Health Service on the incineration of refuse was continued in an effort to obtain information for designing and operating incinerators capable of producing completely innocuous discharges.52/

Preliminary tests with a cylindrical furnace of 2-square-foot grate area were designed mainly to measure the flow of heat and the movement of the ignition plane and combustion into a deep bed of refuse. An incinerator with a 10.6-square-foot grate area and an air lock for periodic feeding of bags of refuse was used in subsequent tests. The refuse fuel used consisted of a mixture of leafy vegetables, sawdust, and cardboard and newspaper, with a moisture content of 25 percent.

In the larger incinerator combustion temperatures could be maintained when the air rate was 80 c.f.m. and a 3-pound bag of fuel was fed into the incinerator every 2 minutes. Oxygen in the flue gas varied from about 4 to 7.5 percent, dropping rapidly to a minimum after each fuel addition or disturbance of the fuel bed. Luminous flame and smoke appeared in the stack with each fuel addition. Studies are now in progress to determine requirements for burning volatile matter and the efficiency at which the incinerator volume is used.

Air Pollution by Sulfur Dioxide

Air pollution is a serious problem in many highly populated urban areas of the United States. About 21 million tons of sulfur dioxide are emitted

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52/ Corey, R. C., Orning, A. A., Schwartz, C. H., and Pfeiffer, J. J., A Progress Report on the Experimental Studies of Incineration by the U.S. Bureau of Mines: Proceedings, Symposium on Developments in Incinerator Research, May 23, 1957, published by New York University College of Engineering, Research Div., January 1958, pp. 31-58.

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to the atmosphere per year, more than 40 percent of it produced in the generation of electric power. As the availability of low-sulfur fuels decreases and demands for electric power increase, the sulfur dioxide emitted to the atmosphere will increase. This pollutant is irritating to the respiratory tract, deleterious to plant life, and corrosive to metals and other building materials. In cooperation with the Public Health Service, the Bureau is investigating methods for decreasing air pollution resulting from sulfur dioxide in flue gases and unburned hydrocarbons in automotive exhaust.

Several regenerative processes have been developed for removing 0.05- to 0.3-percent sulfur dioxide from the flue gases of power plants. In an effort to improve these techniques, the Bureau continued to investigate hot removal techniques, including (1) absorption by solid oxides, (2) absorption by impregnated charcoals, and (3) catalytic oxidation. Attention is also being directed towards the development of a method of regenerating the spent solids used in sorption techniques, to permit further use and recovery of sulfur in a form suitable for manufacture of sulfuric acid. Published information was reviewed on the chemistry of sulfur dioxide, with emphasis on reactions having current and potential applications to the problems of air pollution.53/

A comprehensive screening program was undertaken for testing solids as absorbents, and oxidation catalysts. A simulated flue gas with 0.3 percent sulfur dioxide was used in tests conducted at an hourly space velocity of 1,050 and 265° and 625°F. The most active preparations were oxides of manganese, cobalt, copper, and aluminum. Their regeneration is under study. An alkalized alumina was found to remove sulfur dioxide quantitatively from the simulated flue gas at both temperature levels. The spent absorbent was regenerated repeatedly by hydrogen, carbon monoxide, or producer gas, and sulfur was recovered as hydrogen sulfide and elemental sulfur. The effectiveness of the regenerated oxide equalled that of the original preparation. Activated carbons did not absorb sulfur dioxide unless they were impregnated with metallic oxides, such as copper and chromium oxide. Since the pure metallic oxides are more effective and should be easier to regenerate, studies of activated carbons were discontinued.

An alkalized vanadia catalyst supported on silica gel converted substantial amounts of sulfur dioxide to trioxide at temperatures available in power plant practice. As an oxidation catalyst does not undergo chemical change and would not have to be regenerated frequently, and as sulfur trioxide may be removed by electrostatic precipitation, this technique has potential application and is being studied further.

Air Pollution by Automobile Exhaust

Unburned hydrocarbons in automotive exhaust contribute significantly to air pollution in some urban areas, but might be controlled by catalytic oxidation. The development of catalysts is being investigated; a series of 21 oxides were tested with a number of hydrocarbons.

53/ Bienstock, D., Brunn, L. W., Murphy, E. M., and Benson, H. E., Sulfur Dioxide--Its Chemistry and Removal From Industrial Waste Gases: Bureau of Mines Inf. Circ. 7836, 1958, 96 pp.

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Tests indicated that branched hydrocarbons are more difficult to oxidize than normal hydrocarbons; low molecular weight hydrocarbons are more difficult to oxidize than those of higher molecular weight; paraffins are more difficult to oxidize than olefins or acetylenes; and aromatic hydrocarbons are more difficult to oxidize than alicyclic and aliphatic hydrocarbons.

Supported and promoted catalysts are now being tested. The most promising preparations will be tested for effectiveness with a one-cylinder engine.

CARBONIZATION AND ITS PRODUCTS

Thermal Decomposition of Coal

Knowledge of tar formation and its reactions will lead to a better understanding of the mechanism of coal carbonization. Different methods of determining the primary thermal decomposition products of coal are being investigated to ascertain the mechanism of coal carbonization. Preliminary investigations of the carbonization of Pittsburgh-bed coal at various temperatures and pressures indicate that the maximum reaction rate for the change from coal to semicoke occurs simultaneously with the development of maximum fluidity and maximum product evolution. Studies are now in progress to establish the interval within the plastic range (350-500°C) at which the reaction rate reaches a maximum.

In another study, the yields and composition of products obtained from the carbonization of coal in a fluidized bed were determined before and after thermal cracking. Data obtained from thermal cracking at various temperatures and with different concentrations of tar vapors may provide an indication of the changes that occur when tar vapors pass through the coke oven. The effect of bed temperatures, pretreatment of the coal, and concentration of vapors in the carbonizer on the yields and quality of the vapors that enter the cracker have been determined. The pitch content of the vapors increased with increasing bed temperature. The tar distillate increased by 2.4 gallons per ton of coal and the pitch decreased by 3.7 gallons when a coal was preheated before carbonization to slightly below its fusion point. Less gas and vapor were obtained when the superficial steam velocity was increased and the coal feed rate decreased. When coal was blended with steam-dried lignite instead of char, the tar was less viscous, gas and light-oil yields increased considerably, but the pitch yield remained about the same. The amounts of products from coal and lignite carbonized separately are not the same as when a mixture of the two is carbonized; vapor-phase reactions between the products of each evidently occur when a mixture of the two is carbonized.

The temperature of the thermal cracker had a significant effect on the extent of the cracking. Tar yield and quality were not significantly affected when tar vapors produced at 550°C. were cracked at 600°C. However, when the cracker temperature was 700°C. the tar distillate was reduced from about 12 to 5 gallons per ton, whereas the pitch was reduced only from about 25 to 23 gallons per ton. The residence times of the vapors in the cracker were about 1 second. Yields of tar distillate and pitch were reduced still further when the cracker temperatures were increased.

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Expansion During Carbonization

The expansion of coal during coking is of particular interest to coke-oven operators because of the danger of oven damage from coals or blends that expand excessively during coking. Expansion tests have been made in the sole-heated oven as part of the survey of carbonizing properties of American coals. Formerly, only coals containing more than 65 percent dry, mineral-matter-free fixed carbon were tested. However, as lower-rank coals have a major influence on the expansion of a blend in which they are the major component, a knowledge of their expanding tendencies appeared desirable. In 1958, 19 coals from Pennsylvania, West Virginia, and Alabama were tested. All except four coals contracted and would be safe for oven coking, but the expanding coals would require blending with high-volatile contracting coal.

Coals containing high percentages of ash are usually cleaned before carbonization to improve the quality of the cokes. The removal of ash, however, also affects the expanding tendency of coal. For example, Pittsburgh coal containing 10.1 percent ash contracted 13.6 percent in the sole-heated oven; when the ash was reduced to 5.3 percent, contraction was reduced to 5.5 percent. Thus, under centain conditions a coal which might be safe for coking with its original ash content may become dangerously expanding after cleaning.

Effect of Preheating on Carbonization

To determine whether preheating coals before carbonization would improve their coking properties, three coals and a blend of two coals were preheated to temperatures below the fusion range before testing in the Bureau's BM-AGA pilot oven.54/ The effects of preheating on the chemical and physical properties of the coals, yields of carbonization products, and strengths of cokes produced were determined. Weaker coking coals were improved appreciably as a result of preheating, whereas the more strongly coking coals and blends showed little effect. Direct charging of preheated coal to the coke oven without cooling gave stronger coke, increased yield of tar, and decreased yield of gas.

In another study, the effects of preheating and oil treatment on the coke produced from a blend of weakly coking high-volatile B bituminous coal (Hiawatha, Carbon County, Utah) with 20 percent of medium-volatile bituminous coal (Wilkenson, Pierce County, Washington) were investigated. Fifty-pound charges of the blend were preheated at 410°F., under nonoxidizing conditions and oil treated with standard coal-spray oil, at the rate of 1/2 gallon per ton in a 10-inch cylindrical retort. Coke quality was not improved by preheating, regardless of whether the preheated coal was charged hot or cold. Howevery, the oil treatment (with preheat and hot charging) resulted in larger coke with about the same resistance to tumbling as the coke produced by preheating alone.

54/ Smith, F. W., Birge, G. W., Wolfson, D. E., and Reynolds, D. A., Better Coke by Thermal Pretreatment of Coal: Results for Illinois No. 6-, Pittsburgh-, and Mason-Bed Coals: Bureau of Mines Rept. of Investigations 5418, 1958, 26 pp.

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Effect of Cleaning on Carbonization

The effect of cleaning on the carbonizing properties of coals was investigated with uncleaned and cleaned coal obtained by a gravity separation, in which the ash contents were reduced approximately 50 percent. Six coals with 18.7 to 5.2 percent original ash content were tested. Cleaning of the coals that contained more than 10 percent ash caused an appreciable increase in the 1-inch and 1/4-inch tumbler indexes of cokes carbonized in BM-AGA retorts at 900°C. The 1-1/2-inch shatter index was only slightly improved. When the float-and-sink coal fractions were recombined in the proportions present in the original coal, and carbonized, the cokes produced had strength indexes similar to those from the run-of-mine coal; thus indicating that the organic liquid used in the cleaning process had no effect on the carbonizing properties of the coals.

Effect of Size Distribution on Carbonization

In a continuing investigation of the effect of size distribution on fluidity developed in coking and on the resultant coke, blends of coal of various controlled sizes and a single inert were carbonized.

Effect of size distribution on coking characteristics was determined on Upper Freeport coal, a low-volatile bituminous coal of low fluidity. The coal crushed below 20-mesh was screened into different fractions, and the abrasion indexes of the chars were obtained. The abradability appeared to be independent of size in this range. No significant changes in volatile matter and ash content were noted in fractions, indicating that the coal composition was the same for all sizes. However, the coking characteristics of the coal fractions differed considerably when blended with 40 percent by weight of finely pulverized coke. The strength of the carbonized residues increased progressively as the screen fractions became finer. Furthermore, the product of a blend, prepared by recrushing the minus 20- plus 35-mesh sample until the material passed through a 150-mesh sieve, had a much higher abrasion index than that of the original coarser fraction from which it was prepared. This phenomenon probably results from more intimate contact between the coal particles and the inert material.

Comparison of Commercial and Experimental Cokes

The physical properties of three Canadian industrial cokes were compared with those of similar cokes produced by two laboratory methods--that is, by the BM-AGA method used by the Bureau, and the 25-pound Bethlehem sole-heated oven used by the Canadian Department of Mines.[55] Results of ASTM shatter and tumbler methods on BM-AGA and industrial cokes agreed closely except for the 1-inch tumbler test of one coke obtained by carbonization industrially at the

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55/ Swartzman, E., Botham, J. C., Tibbetts, T. E., Burrough, E. J., and Birge, G. W., A Study of the Relation Between the Properties of Cokes Produced by Means of Two Laboratory Methods and Coke Produced Industrially: Department of Mines and Technical Surveys, Ottawa, Canada, Rept. 1R 58-138, August 1957, 68 pp.

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relatively slow coking rate of 0.73 inches per hour, as compared with 1 inch per hour for the other industrial cokes.

Effect of Oven Width on Carbonization

Oven widths for experimental and commercial slot-type ovens differ appreciably in most instances, the former usually ranging from 10 to 14 inches and the latter from 14 to 21. Application of results obtained with experimental ovens to commercial practice thus requires extrapolation and, since only limited information is available on the effect of oven width, such extrapolation cannot be made with confidence. Information on the effects of oven width would also be helpful in selecting the optimum width for commercial slot-type ovens. An investigation has been directed toward obtaining such information.

The Tuscaloosa oven was adapted to permit operation at three different widths.[56] Because changes in oven width at any given flue temperature are accompanied by changes in coking rate, the effect of changes in oven width on the properties of the cokes produced must be considered. As a first step in analyzing the data, coking times, flue temperatures, bulk densities, and oven widths were correlated by a least-squares method. Although the effects of variations in flue temperature and bulk density on coking time were known, the effects of oven width had not been determined previously for the Tuscaloosa oven. The term "coking time" in this study refers to the minimum time for carbonizing and makes no allowance for soaking or tempering.

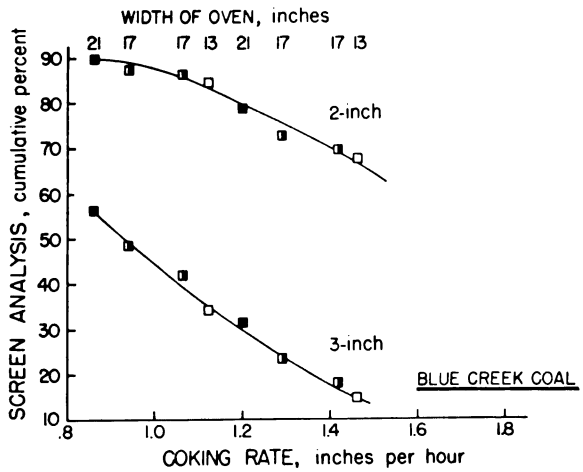

FIGURE 7. - Effects of Changes in Oven Width and Coking Rate on Plus- 2- and 3-Inch-Screen Indexes for Tuscaloosa Oven Cokes.

To evaluate the relationships between coke quality, oven width, and coking rate (Figure 7), physical tests were made on cokes produced at different rates and 3 oven widths--21, 17, and 13 inches--in tests at two or more flue temperatures, including 2,000° and 2,300°F. The results indicate that these changes in flue temperature and oven width had little or no effect on the properties of cokes produced at constant coking rate.

Coking Properties of Western Coals

The cooperative research program on improving the quality of coke made from Western high-volatile bituminous coals, begun in 1956, was concluded in 1958. The study included a total of 52

56/ Gayle, J. B., and Eddy, W. H., Carbonizing Tests With Tuscaloosa Oven-Studies of Oven Width, Flue Temperature, and Coking Rate: Paper presented at AIME Blast Furnace, Coke Oven, and Raw Materials Conference, April 14-16, 1958, Cleveland, Ohio, Proceedings AIME, vol. 17, 1958, pp. 193-208.

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high-temperature carbonization tests in the 500-pound, 14-inch, slot-type coke oven. A study of the effects on bulk density of pulverization, surface moisture, oil type and concentration, and storage was also made.

Slot-oven tests were made on blends of four Utah and Colorado high-volatile bituminous coals and two medium-volatile bituminous coals from Colorado and Oklahoma to study the effect of coking at rates ranging from 0.56 to 1.35 inches per hour. Slower coking resulted in larger, denser coke that was more resistant to breakage by shatter and by tumbling, while faster coking rates favored the production of a minimum of breeze, as indicated by the tumbler hardness index.

Data obtained in a study of the effects of preheating coke-oven charges to 500° F. indicated the following trends:

1. Preheating the coal blend with or without oxidation resulted in smaller, less dense coke. However, such coke was more resistant to breakage from tumbling. Little or no effect was observed on resistance to shattering.

2. Pulverization from a nominal size of 3/8 by 0-inch to 80 to 85 percent through 1/8-inch resulted in decreased bulk density and increased coking rate.

3. Addition of one-half gallon spray oil per ton of coal to two samples resulted in an increase in bulk density and a decrease in coking rate.

Interstation Coke Comparison and Standardization

Studies of the carbonizing properties of coals and the quality of metallurgical cokes have been conducted for several years by three Bureau stations (at Pittsburgh, Pa., Tuscaloosa, Ala., and Denver, Colo.) representing regions of the country in which coke production is of major interest. Ovens of different size and shape were used at the three stations. To establish the effect on coke quality that such differences might cause, the three stations made simultaneous tests on replicate samples of four coals or coal blends.

Standard ASTM tests were used to determine the chemical and physical characteristics of the cokes, and preliminary review of results on the first three samples indicates a high degree of quantitative agreement among the stations. The close agreement in the physical properties of the cokes from the three sources indicates that the shape and size of the coking chambers had little effect on the quality of the coke produced.

Survey of Carbonizing Properties of Bituminous Coals

The survey of the carbonizing properties of coals from the principal American coal beds was continued with tests of 17 Pennsylvania and 13 West Virginia coals. The Bureau of Mines-American Gas Association (BM-AGA) apparatus was used to carbonize approximately 200-pound samples at 900° C.; yields and properties of coke and other products were determined. Recently, attention has been directed toward sampling marginal areas where, although mining has not been extensive, minable reserves exceed 50 million tons in a given county. The availability of such data has benefited small operators.

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Allegheny County, Pa.

Coke produced from the Redstone bed, sampled at two locations in the county, had satisfactory strength for metallurgical use; however, the ash content of both samples was moderately high, and one of them contained an excessive amount of sulfur.

Westmoreland County, Pa.

Coke from two samples of Redstone coal in this county had lower strength indexes than coke from the same bed in Allegheny County. The chemical properties of these samples do not meet present metallurgical standards.

Greene County, Pa.

Three coals, representing the Pittsburgh and Waynesburg beds, had sulfur and ash contents ranging from 1.5 to 3.2 and 9.2 to 16.9 percent, respectively. The Pittsburgh-bed sample had chemical and coking properties acceptable for coking-coal blends. Coke from Waynesburg coals had lower 1/4-inch tumbler indexes than the average for coals of high-volatile rank, but they probably could be improved to meet metallurgical requirements by cleaning.

Jefferson County, Pa.

Two coals representing the Upper Freeport and Lower Kittanning beds yielded cokes with moderate strength indexes. Although sulfur and ash in each sample slightly exceeded desirable limits, these coals could be used in coking blends with coals that have more satisfactory chemical properties.

Clarion County, Pa.

Two samples of the Clarion bed ranked low in the high-volatile A classification and yielded cokes with very low tumbler indexes. Each coal contained 4.0 percent or more sulfur. Poor quality and unsatisfactory chemical properties of the cokes make those coals unsuitable for metallurgical use. As could be expected on the basis of their comparatively low rank, the tar yields (19.2 and 22.1 gallons per ton of coal) were relatively high for bituminous coking coals.

Clearfield County, Pa.

Three samples from the Middle Kittanning and Brookville beds ranked high in the medium-volatile classification and yielded cokes with excellent strength characteristics. Because sulfur ranged from 2.5 to 3.7 percent and the ash contents were moderately high, these coals are less desirable for coking purposes than most coals presently used in the Appalachian region. However, owing to their very strong coking tendencies they could be desirable additions, in minor proportions, to metallurgical coking blends.

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Fayette County, Pa.

Although Upper Freeport coal from one mine was medium volatile in rank and yielded very strong coke, the sulfur content of 3.0 percent would limit the use of this coal for production of metallurgical coke.

Washington County, Pa.

A sample of the Waynesburg bed contained 3.3 percent sulfur and 16.8 percent ash. The moderate strength of the coke could be improved appreciably by reducing the ash content. However, it is doubtful whether the sulfur content could be reduced enough to permit use as a major component of metallurgical blends.

Fayette County, W. Va.

No. 2 Gas and Powellton coals were high-volatile A with very good coking properties; sulfur was less than 1.0 percent, and ash was also low. The strength indexes of their cokes were about average. These coals would be satisfactory as major components of coking blends. Fire Creek coal was medium volatile in rank and coked very strongly. All of these coals would be satisfactory for metallurgical use. A sample of Winifrede coal from the border of Kanawha-Fayette Counties was chemically suited for metallurgical use but yielded weak, fingery coke.

Boone County, W. Va.

No. 5 Block coal was satisfactory in all respects for use in coking-coal blends. Although the dry mineral-matter-free fixed-carbon content of each coal exceeded 65.0 percent, samples of Lower Winifrede and Upper Winifrede yielded very weak coke. In the Boone County area coals of this rank usually coke more strongly. The low coke-strength indexes of these coals may therefore be attributed to weathering of the coal in the prospect openings from which the samples were taken. The low yield of tar, about 8 gallons per ton of coal, also supports this explanation. When the Lower Winifrede coal was blended with 20 percent medium-volatile Fire Creek coal from Fayette County, the coke-strength indexes increased appreciably but not enough to meet present metallurgical requirements.

Marion County, W. Va.

Five samples, representing the Sewickley, Waynesburg, and Pittsburgh beds, were relatively low rank with dry, mineral-matter-free fixed carbon, ranging from 56.0 to 60.6 percent. With the exception of one Pittsburgh-bed sample, all contained excessive amounts of sulfur and two also contained excessive ash. The Pittsburgh-bed coal came from the northern part of the county and was relatively low in sulfur and ash content. Coking properties were average as compared with Appalachian coals of similar rank. The Waynesburg sample was the strongest coking, and cleaning may upgrade it for metallurgical use.

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McDowell County, W. Va.

Fifteen coals from McDowell County representing 13 beds were carbonized and their carbonizing properties were evaluated.57/ Most of the coals had satisfactory chemical properties as mined; the others could be upgraded without difficulty to meet present standards for metallurgical coking coals. The dry, mineral-matter-free fixed-carbon content of the coals ranged from 64.4 to 84.2 percent. Four were high-volatile A, three were medium volatile, and eight were low volatile. Coke strength ranged from good to excellent, according to rank. However, the medium- and low-volatile coals expanded in the sole-heated oven and would require blending with lower-rank coals for industrial carbonization.

Floyd County, Ky.

Samples representing the Elkhorn Nos. 1, 2, and 3, and Fireclay beds were carbonized by the BM-AGA method, and results indicate that Floyd County coals would be suitable for industrial carbonization only when blended with appropriate proportions of higher-rank coal.58/ Except for the 1/4-inch tumbler indexes, which were relatively high, strength indexes of the cokes were usually low compared with those of cokes from other Appalachian coals of similar rank. However, blends containing 20 percent Pocahontas No. 3 low-volatile coal indicate that the blending properties of the Elkhorn coals are equivalent to those of high-volatile coals from the Pittsburgh district.

Metallurgical Use of Anthracite

Calcination

Research was continued on a process for producing thermally stable anthracite by calcining it in a vertical-shaft retort. The tests have shown that certain anthracites are more sensitive to decrepitation than others. This tendency appears to become less with decreasing volatile-matter content of the feed material. Preheating the charge resulted in increased throughput rates and yields of large-sized product. Both counter- and co-current direct preheating and countercurrent indirect preheating were tried. Only direct countercurrent flow proved satisfactory for heating.

The calcined anthracite has been tested in three foundries; two of the foundries had previous experience with anthracite and encountered no difficulty in its use. In both installations, metal temperatures and melting rates obtained with calcined anthracite were superior to those obtained with their normal foundry fuel. However, since iron specifications in neither foundry are stringent, additional tests were begun in a third foundry, which specializes in high-quality merchant production.

57/ Birge, G. W., Naugle, B. W., and Wolfson, D. E., Carbonizing Properties of McDowell County, W. Va., Coals: Bureau of Mines Rept. of Investigations 5432, December 1958, 15 pp.

58/ Wolfson, D. E., and Birge, G. W., Carbonizing Properties of Floyd County, Ky., Coals: Bureau of Mines Rept. of Investigations 5399, May 1958, 13 pp.

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Briquetting

Briquets containing approximately 82 percent anthracite, 10 percent bituminous coal, and 8 percent of 70° C. m.p. coal-tar pitch, were formed at approximately 3,000 p.s.i. and calcined at 1,750° F. for 3 hours. These briquets showed a higher ASTM tumbler stability than most premium cokes. Recent tests have shown that superior briquets can also be produced from mixes without bituminous coal; however, the pitch content must be increased to 10-12 percent. Other coal-tar pitches and asphalt-residue binders have also been tested.

A study directed toward determining the most suitable anthracite sizes for calcination and briquetting indicated conventional Buckwheat No. 5 (through 3/64-inch screen by 0) to be best.

When bituminous coals are used, a high-volatile coal having a high fluidity and a free-swelling index of at least 6.5 is required. The free-swelling index appears to correlate best with the strength of briquets made from mixes containing bituminous coal.

A pilot briquetting-plant with a capacity of approximately 1-1/2 tons per hour and a coal drier were designed and built during the year. Tests were begun to develop information on the economics of the process, and to prepare enough briquets for tests in commercial metallurgical equipment.

Preliminary tests with the vertical-shaft equipment have indicated that the agglomerated product could be completely calcined without difficulty. However, the volatile products resulting from the coking of the pitch and bituminous coal components could not be completely exhausted and pressure build-up in the upper section of the retort made continuous operation impossible. This condition may be alleviated by use of larger exhaust equipment.

Low-Temperature Carbonization

A small-scale carbonization assay test, originally reported in 1953, has been used for a wide variety of coals and has proved to be useful both as a standard bench-scale test and as a research tool in carbonization investigations. Improvements now make this test applicable to medium and high temperatures.59/

Western Coals

The production and evaluation of industrial carbons from noncoking or slightly coking coals are a part of the Bureau's program concerned with upgrading Western U. S. coals. A special study was made of lignite from an area about 10 miles north of North Dakota, in the Province of Saskatchewan, Canada. A 5-1/2-ton sample was crushed to 1/4 by 0-inch, and dried from 35.2 to 7.2

59/ Goodman, J. B., Gomez, Manuel, and Parry, V. F., Laboratory Carbonization Assay of Low-Rank Coals at Low, Medium, and High Temperatures: Bureau of Mines Rept. of Investigations 5383, 1958, 49 pp.

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percent moisture by entrainment in hot inert gas. Although assay results indicated that maximum carbonization yield would occur at 550° C., the gain from 500° to 550° C. was comparatively small, and the pilot-plant test was therefore made at the lower temperature. About 6.5 gallons of tar plus light oil and 926 pounds of char were produced per ton of raw lignite, after allowing for fuel requirements of the drying operation.

A second test was made with Sweetwater (Sweetwater County, Wyo.) high-volatile bituminous coal, in the vertical gravity-flow carbonizer designed for the Indian lignite project, to study the possibility of producing a char that could be substituted for coke breeze in phosphate ore reduction. The Sweetwater coal sized at 1-3/8-inch, was carbonized to 1,380° F., but the char produced was weaker than the coke breeze now used at Westvaco, Idaho, for phosphate ore reduction.

Indian Lignite

A program of technical assistance to the Indian Government in connection with the utilization of a low-grade lignite from Neyveli, near Madras, was completed this year. Its objective was to develop a smokeless, easily ignitible domestic fuel and to provide a pilot plant in which substantial quantities of lignite could be processed and operating variables studied. The plant, with its associated laboratory facilities, was also to be used to train Indian technologists. A secondary objective was production of coal tar for further study by Indian chemists.

Early in 1958 the plant was erected at Neyveli by the Indian Government, under the supervision of the fabricator, Silver Engineering Works. Bureau engineers supervised initial operation, reviewed results, and conferred with Indian technologists on plans for additional research with the facility.

The operation comprises four major steps: Crushing, drying, briquetting, and carbonizing. Design capacity of the first three units is 2.5 tons of raw lignite per hour; of the carbonizer, 500 to 1,000 pounds per hour.

Initial operation of the plant was handicapped by difficulty in obtaining reasonable quantities of the type of raw lignite for which the process was designed. Production from the open-pit mine is scheduled to begin in 1960, and several lignite development projects will compete for the small quantities available from test pits. A supply of lignite bore-hole cuttings was available for experimental use but was physically unsuitable. When normal raw lignite was obtained and used, smooth operation and a satisfactory product resulted.

Low-Temperature Tar

Two of the Bureau's programs are concerned with the investigation of low-temperature tar. One program is directed to a study of lignite tar and tar from subbituminous coals, the other deals mainly with tar from bituminous coal. This comprehensive attack should facilitate the increase in low-temperature tar value and contribute to the economic development of low-temperature carboni-

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zation. The potential utilization of some of these tar products was described in recent publications.60/

Tar From Lignite

The development of lignite as a commercial material is an important factor in the future industrialization of the north-central states where lignite represents a great natural resource. A program to provide data needed to develop the maximum value of liquid carbonization products and to establish a basis for industrial utilization of such products was continued. The present approach in studying lignite tar is that of detailed characterization of fractions obtained in low-temperature carbonization. In a related project recently initiated, assay procedures are being developed that would be particularly adaptable to evaluating primary tars and would supply data required for interlaboratory comparison and commercial evaluation. This work is also being complemented by an investigation of humic acids, which are available in quantity from naturally or artificially oxidized lignite and may be a significant factor in the commercial development of lignite.

The major effort has been concentrated on tar acids (phenolics), potentially valuable but unstable components of the tar. During the year, narrow-boiling tar-acid fractions in the range of 234° to 268° C. were separated and characterized. To date 29 different phenolic compounds including a number of pyrocatechols and guaiacols,61/ have been identified in the lower-boiling fractions of the tar. These compounds appear to be characteristic of North Dakota lignite low-temperature tar.

In five exploratory experiments lignite low-temperature tar was hydrogenated at 3,000 p.s.i.g. hydrogen pressure and examination of the products begun. The high-boiling creosote oil and the whole tar hydrogenated readily, and preliminary material balances indicate liquid-product yields of 90 to 95 weight-percent. The proportion of tar acids, tar bases, and neutral oil remained essentially unchanged, although a shift within these groups toward lower-boiling materials was noticed. The amount of pitch produced on distillation was reduced markedly. Results indicate that complete conversion to hydrocarbons might be accomplished with a high percentage of aromatics.

Another project was concerned with the thermal solution of oxidized lignite in a partly-saturated hydrocarbon (tetralin). Preliminary tests indicate that pretreatment, partial hydrogenation, and subsequent solution of the lignite may be feasible.

In developing an assay method for low-temperature tars, known procedures that could be adopted were explored and attempts were made to avoid distillation if possible. The solvents and the conditions required to isolate fractions that may be useful for commercial development or in interlaboratory comparison are being investigated.

60/ McGee, J. P., Coal's Place in the Space Program: Coal Utilization, vol. 12, No. 9, September 1958, pp. 31-33.

61/ Beckering, Willis, and Fowkes, Walter W., Analysis of Polyhydric Phenol Mixtures: Anal. Chem., vol. 30, No. 8, August 1958, pp. 1336-1338.

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Lignite low-temperature tar can be separated into several fractions by selected solvent combinations. Fractionation seems to depend on molecular weight or chemical character. The development of efficient separations would permit avoiding the changes induced by thermal treatment of primary tar.

Tar From Bituminous Coal

Maximum yields of tar are obtained at relatively low carbonizing temperatures, about 500° C. As a result of recent developments in low-temperature carbonization, especially with continuous, entrained-state techniques, greater industrial attention has been directed to the properties of the tar products. Credits derivable from this product are now regarded as the key to the economic development of low-temperature carbonization. The potentialities of low-temperature tar as a source of chemicals are being explored by different research groups, particularly in Europe. A critical review of low-temperature tar chemistry, summarizing existing knowledge on tar formation and alteration, was published.62/ Tar yield and character appear to be related to the rank and oxygen content of the coal carbonized. The effect of carbonizing conditions has also been studied. Carbonization assays have been made on a wide variety of coals.

Research on the composition of a sample of low-temperature bituminous coal tar progressed to the point where a total of approximately 150 individual aromatic compounds were identified, and the amounts of most of these compounds were quantitatively determined. These compounds all fall in 3 classes: (1) tar acids (phenols, indanols, and naphthols); (2) tar bases (pyridines, quinolines, and anilines); and (3) aromatic hydrocarbons (benzenes, indanes, indenes, tetralins, naphthalenes, and biphenyls).

Some conclusions may be possible concerning the composition and treatment of low-temperature tar, at least with regard to the lower-molecular-weight aromatic compounds: (1) The distribution of isomers in the tar can be compared with the distribution expected from thermodynamic equilibrium. By this means some fundamental information can be obtained about the carbonization process and possibly about coal structure. (2) A rational approach can be taken toward certain aspects of refining tar. For example, only 20 of 150 identified aromatic compounds are free of alkyl groups and partly hydrogenated fused rings that would be equivalent to alkyl groups. Thus, low-temperature tar is more similar to crude petroleum oils than to coke-oven tar. (3) A new approach can be taken toward utilization of low-temperature tar, taking advantage of special classes of compounds now known to be present. For example, 11 indenes, styrenes, and benzofurans are present in significant quantities. These compounds contain a highly reactive olefinic bond and form a variety of copolymers with each other when dilute solutions of the compounds in their natural distillate fractions of the neutral oil are treated.

Countercurrent distribution fractionation was used to separate the higher boiling phenols, about 100 fractions being obtained from each tar-acid distil-

62/ Gomez, Manuel, Goodman, J. B., and Parry, V. F., General Properties of Low-Temperature Tar: Bureau of Mines Bull. 569, 1958, 31 pp.

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late cut, and gas-liquid chromatographic fractionation was used to separate the low-boiling phenols.[63] A tar-acid mixture boiling up to 234° C. was analyzed without recourse to time-consuming procedures.[64] An ultraviolet spectrophotometric method was developed for determining total pyridines and total quinolines in coal-tar distillates.[65] A spectrophotometric technique was also used for analyzing a typical low-temperature bituminous-coal-tar base mixture boiling up to about 355° C.[66] A total of 51 individual compounds was identified in the tar-base mixture and their quantities estimated. This work constitutes the first fairly comprehensive analysis of low-temperature bituminous-coal-tar bases; more than 80 percent of the compounds described had not been identified before.

Gas-liquid chromatographic fractionation was used to separate the lower boiling aromatic hydrocarbons, based on the distribution between an immobile liquid phase (Apiezon L grease supported on 30- to 60-mesh fire brick) and a mobile gas phase (helium) at 150° C.

Upgrading and Utilization

A process for isolating tar acids with monoethanolamine as the extractant was reported in 1956, and an alternate procedure for recovering the tar acids from the monoethanolamine solution was discussed in 1957. In the original procedure, carbon dioxide was used to spring the phenols from the amine solution. A method has now been developed for titrating the amine carbonate solution.[67] In the alternate procedure, the amine solution was diluted with water, and the tar acids were extracted with a volatile solvent such as benzene. With this procedure, a tar acid of improved quality was obtained. After repeated recycling, the monoethanolamine showed some reduction in selectivity; accumulation of dark-colored materials of unknown composition may make it necessary to redistill a part of the monoethanolamine to reduce the build-up of impurities. In the continuous extraction of tar acids from a 150° to 350° C. fraction of tar distillate oil, about 80 volume-percent of the tar acids was extracted when the volume ratio of amine to tar acids was 2:1.

63/ Karr, Clarence, Jr., Brown, P. M., Estep, P. A., and Humphrey, G. L., Identification and Determination of Low-Boiling Phenols in Low-Temperature Coal Tar: Anal. Chem., vol. 30, No. 8, August 1958, pp. 1413-1416.

64/ Karr, Clarence, Jr., Brown, P. M., Estep, P. A., and Humphrey, G. L., Analysis of Low-Temperature Tar Phenols Boiling up to 234° C.: Fuel, vol. 37, No. 2, April 1958, pp. 227-235.

65/ Chang, T. L., and Karr, Clarence, Jr., Ultraviolet Spectrophotometric Determination of Total Pyridines and Quinolines in Low Temperature Coal-Tar Distillates: Anal. Chem., vol. 30, No. 5, May 1958, pp. 971-972.

66/ Karr, Clarence, Jr., and Chang, T. L., Spectrophotometric Analysis of the Distillable Low-Temperature Tar Bases: Jour. Inst. Fuel, vol. 31, No. 215, December 1958, pp. 522-527.

67/ Chang, Yi-Chung, Potentiometric Titration of Free Amine and Amine Carbonate in Carbonated Monoethanolamine Solutions: Anal. Chem., vol. 30, No. 6, June 1958, pp. 1095-1097.

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The value of the tar acids extracted from distillate oil fractions boiling up to 350° C. would be much enhanced by increasing the concentration of lower boiling phenols in the product. In the low-temperature tars investigated, at least 75 percent of the tar acids boil above and fall outside of this desirable range. Cracking of alkyl side chains and other groups attached to aromatic rings would increase the concentration of lower boiling phenols. Studies of dealkylation were therefore continued.

The laboratory dealkylation studies of phenols were undertaken under a cooperative agreement with the Chemical Engineering Department of West Virginia University. Dealkylations have been carried out at atmospheric pressure over a commercial silica-alumina cracking catalyst. At 750° to 850° F. alkyl groups, especially in the para position, have been cracked to give lower boiling phenols. Data on tar-acid samples show that the lower boiling fractions were more easily cracked to lower boiling phenols than were the higher boiling tar-acid fractions. Isomerization, disproportionation, and cracking of tar acids to gases and carbon were also encountered. Figure 8 shows the flow diagram of the laboratory-scale catalytic dealkylation unit with chromatography attachments so that liquid and gaseous products can be analyzed as they leave the reaction zone.

Small pilot-scale studies on dealkylation have yielded results that qualitatively agree with the laboratory results. Some of the data indicated that benzene included in the tar-acid charge acted as an alkyl group acceptor during dealkylation.

Studies were also continued on separating neutral tar oil fractions into chemical types (aromatic, olefinic, and saturated hydrocarbons) by means of solvent extraction. The aromatic fractions might be used as raw materials for manufacturing chemicals, or after hydrogenating, as high-energy jet fuels.[68] Investigations of selective solvents for aromatics have been limited to three dipropionitriles. Laboratory extractions at room temperature have shown that these dipropionitriles have essentially the same capacities for separating aromatics in the 150° to 200° C. and in the 200° to 250° C. fractions of neutral tar oils. Most of the extracted aromatics were recovered by washing the nitrile extracts with a volatile solvent such as n-hexane or cyclohexane.

GASIFICATION OF COAL

Reaction of Carbon with Steam and Carbon Dioxide

Data on the rate and mechanism of the reaction of steam with coals are basic to the development and design of large-scale coal gasifiers for making synthesis gas or hydrogen. In the course of a study of this reaction, the effect of hydrogen inhibition has been determined with samples (which are suspended from an analytic balance), in a furnace maintained at constant temperature, the rate of weight loss being a measure of the rate of reaction. Gasification was strongly inhibited by low concentrations of hydrogen, the effect decreasing at higher concentrations. Further work is in progress.

68/ Wainwright, Howard W., Coal's Place in Space: Rocket Fuels from Tars: Missiles and Rockets, vol. 4, No. 16, October 20, 1958, pp. 43-48.

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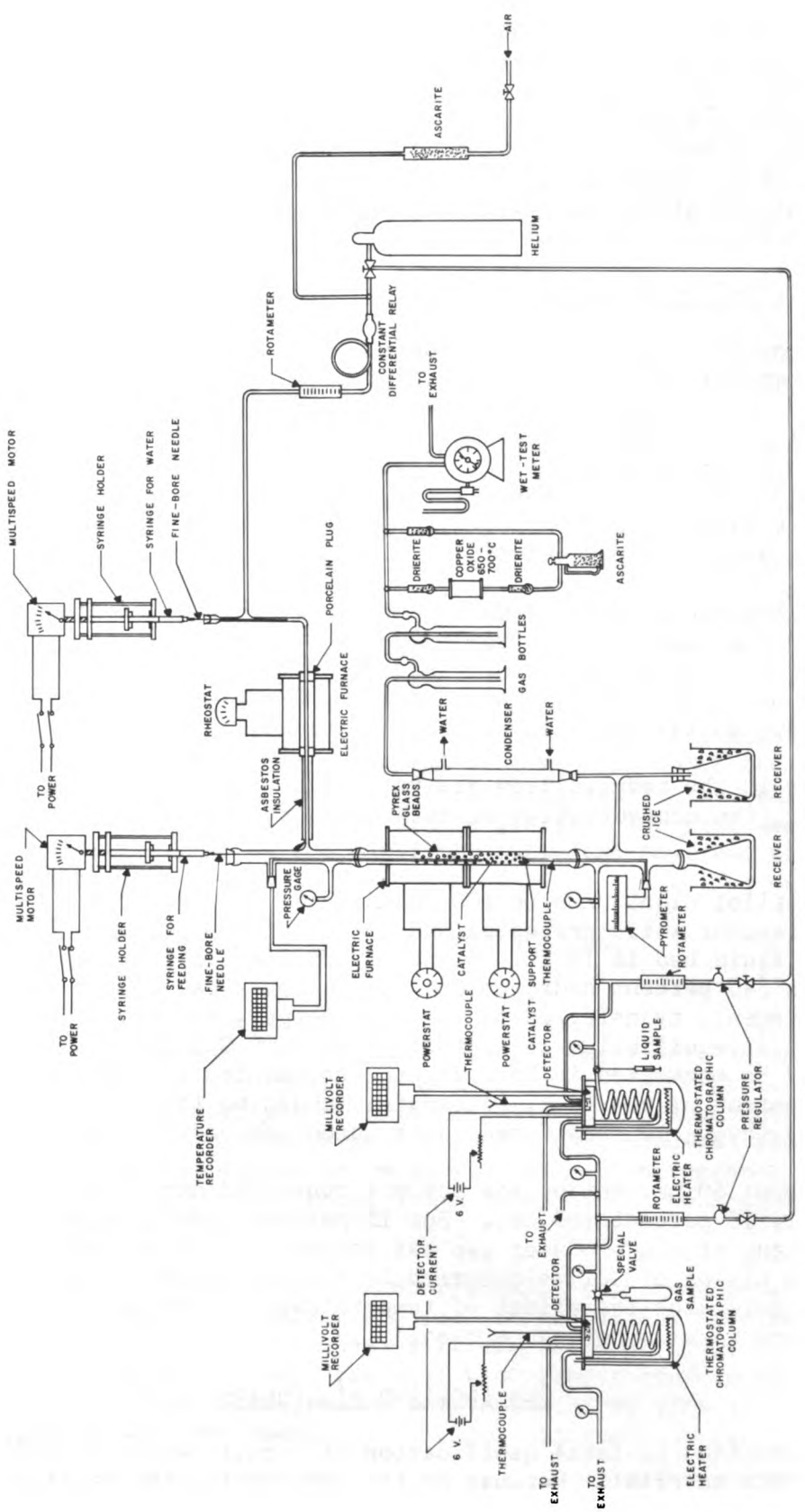

FIGURE 8. - Flowsheet of Laboratory Catalytic Dealkylation Unit With Chromatographic Columns.

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Studies of the oxygen exchange reactions between carbon dioxide and carbon monoxide in the presence of solid carbon were continued. At 700° to 850° C. exchanges from 1 to 30 percent of the theoretical were noted. The exchange mechanism was found to be complex. The pretreatment of carbon has a pronounced effect on the exchange rates; these effects will be studied further. To understand the role of the solid state in the reactions of carbon with oxygen containing gases, an attempt is also being made to calculate the bond energies of oxygen atoms attached to terminal carbon atoms of graphite layers.

Steam-Iron Process for Producing Hydrogen or Synthesis Gas

The production of hydrogen by the reaction of steam and iron has been practiced commercially on a small scale for many years. This process is being investigated as an alternate to the coal-steam-oxygen reaction for converting coal to gas: Iron oxide is reduced by producer gas (derived from coal), and reduced iron is oxidized either by steam to generate hydrogen or by steam and carbon dioxide to produce synthesis gas. If efficient gas utilization and high reaction rates can be achieved, the economics of the two processes for making these gases may be competitive.

In bench-scale studies producer gas containing sulfur compounds has been passed through a fixed bed of iron oxide to study the effect of sulfur on the reactions of iron oxide and iron. Reduction at atmospheric pressure and 800° C. was not significantly retarded by 0.002 to 0.2 percent H_2S, even though the sulfur concentration in the solids increased.

Sulfide in the reduced iron did not affect the formation of synthesis gas; however, the concentration of methane in the product gas dropped from 13 to 0 percent.

In the pilot plant, 20- to 100-mesh solids are fed from a hopper to the top of the reactor while preheated gas enters at the bottom. The maximum height of a fluid bed is 15 ft. Oxidized iron is partly reduced by simulated producer gas (40 percent hydrogen and carbon monoxide, 60 percent carbon dioxide and nitrogen), transferred to the feed hopper, and reoxidized in a subsequent pass. If equilibrium is approached at 800° C., 60 to 70 percent of the gases should be converted in both steps, accompanied by a 30-percent change in the oxide content of the iron, alternately changing the iron to 5 and 35 percent reduced (Fe_3O_4 is considered to be unreduced and Fe completely reduced).

When about 60 percent of the gas was converted during reduction, the solids were only 10 percent reduced. For 15 percent reduction of the solids, only 35 percent of the producer gas was converted. These results suggest that considerable mixing of solids occurred in the fluid bed. Other tests are in progress to determine the effect of temperature, bed height, and gas composition on the conversions of gas and solids.

Gasification of Lignite

Gas production by total gasification of lignite would afford a substantial outlet for this material. Because of its inherently high reactivity, lignite

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appears especially suitable for gasification. A fixed-bed pressure gasification process is being developed for the gasification of lump lignite with a mixture of oxygen and steam. Fixed-bed gasification is characterized by low oxygen requirements and high carbon conversion. In commercial fixed-bed gasifiers ash is removed dry, but theoretical considerations indicate that less steam is needed and gas is produced faster when the gasifier operates under slagging conditions similar to those of a blast furnace. The new gasifier has been designed to operate under slagging conditions, with provisions for conversion to dry-ash removal.

During the past year the gasification pilot plant was 95 percent completed. It is designed so that 3/4 by 1/8-inch lignite will be fed continuously by gravity, from an intermittently charged coal lock and will be gasified, with steam and oxygen introduced into the lower section of the gasifier as it moves downward. The gas stream, leaving at the top, will be cooled, and most of the tar and water vapor from the thermal decomposition and drying of the lignite removed. The molten ash will be discharged from the slagging section into a spray-cooled water bath, quenched, granulated, collected in the slag lock, and removed intermittently (Figure 9). The outer or pressure-bearing wall of the gasifier is cooled by a water jacket; the inside is refractory lined. The slagging section is heavily lined with refractory material to reduce heat losses, and the inner liner, which is exposed to attack by slag at temperatures up to 2,600°F., is lined with silicon carbide. The charge will be supported by a flat silicon carbide bottom with a 2-inch taphole in the center for slag removal. Four injection nozzles, equally spaced around the periphery and 2 inches above the bottom, will admit the oxygen-steam mixture into the gasifier. At present the slagging section is designed for operation up to 100 p.s.i.g.; the rest of the gasifier for pressures up to 600 p.s.i.g. The overall height of the gasifier, including coal and slag locks, is 39 feet 4 inches.

Gasification of Pulverized Coal

Coal-Feeding Methods

In developing equipment for feeding pulverized coal into an entrained-bed gasifier, emphasis has been on operating a coal-water slurry heater. Its design is based on convection rather than radiant heating as originally used, to control the temperature and keep the coal from softening and blocking the coil. Early runs with the new heater were successful, and present work concerns the problem of separating excess steam from the hot coal-steam mixture delivered by the heater.

In a second method (the steam pickup system) for feeding coal continuously to an entrained-bed gasifier, pulverized coal is fed with a star feeder into the stream of superheated steam to the gasifier. At both 150 and 300 p.s.i.g. with steam-coal ratios of 0.6 and 0.3, coal throughputs of 800 pounds per hour were possible in the pilot plant, and attempts are being made to increase the capacity to 1,000 pounds per hour.

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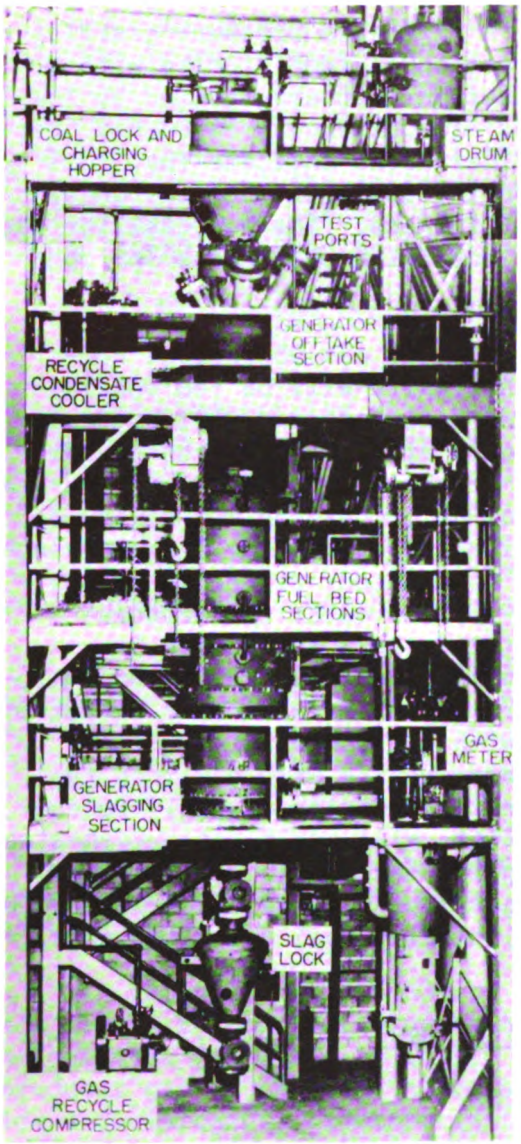

FIGURE 9. - Fixed-Bed Pressure Gasifier, Bureau of Mines, Grand Forks, N. Dak.

Nuclear Energy for Coal Gasification

The use of nuclear fission to supply heat for coal gasification could result in a marked reduction in the cost of synthesis gas from coal. It offers the following advantages: (1) Temperatures are limited only by materials of construction. (2) Since nuclear heat is generated without air or oxygen, it can be used in high-pressure processes without compression costs. (3) No heat is lost through stacks; unused heat is returned to the nuclear reactor. (4) Heat can be transferred at elevated pressures, thereby effecting a substantial increase in heat-transfer economy over heat obtained from combustion processes.

A cooperative program with AEC was therefore inaugurated in 1955 to study the feasibility of using nuclear energy for the gasification of coal.[69/] The indirect process cycle, in which an inert gas, such as helium, is heated in a nuclear reactor up to 2,500° F., and is used as the heat transfer medium, appeared most promising.

Problems were encountered in efforts to design and construct a nuclear field element to operate above 2,000° F. The feasibility of using unclad uranium-graphite fuel elements and helium as a heat-transfer medium is being studied to permit operation at 2,500° F. and 500 pounds per square inch. Finely-pulverized, low-rank coals (subbituminous coals and lignite) had already been gasified in alloy-steel tubes at about 1,800° F. However, higher temperatures would aid the process materially.

Heat Transfer

To obtain information on heat transfer by helium, two units in which helium is circulated at high temperatures have been constructed; one for operation at atmospheric pressure and the other for operation under a pressure of 300 p.s.i.g. The nuclear heat-source is simulated by graphite spheres that are heated by electrical induction. These units are also used to test reactor components.

69/ McGee, J. P., High-Temperature Systems for Nuclear Process Heat, AEC Technical Information Service Extension Rept. TID-7564, December 1958, pp. 305-324.

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In preliminary tests, the atmospheric pressure unit was operated to study the transfer of heat from the graphite spheres to the recycled stream of helium. Data from these tests are being processed. At present in the 250 p.s.i. unit, heat is transferred from helium to a recycled air stream so that operating experience can be gained with a high-temperature, gas-to-gas heat-transfer system, without the added complexity of a gasification system. This latter unit is being tested at 250 p.s.i.g. (Figure 10). Helium is heated to 2,500° F., cooled to 1,000° F. by the indirect exchange of heat with air, and filtered to remove particles that could damage the high-speed compressor that recirculates the helium. The induction-heated simulated reactor is designed for 300 p.s.i.g. Helium enters the top of the vessel at 1,000° F., flows over the graphite elements where the temperature is raised to 2,500° F., and leaves the bottom of the reactor through a refractory-lined pipe leading to the heat exchanger.

Heat transfer was also studied in the laboratory with helium flow through alloy steel and ceramic tubes. Average inside film coefficients were determined at mean surface temperatures from 200° to 1572° F. with helium inlet temperatures ranging from 1,450° to 2,650° F. The data were correlated by an empirical equation.

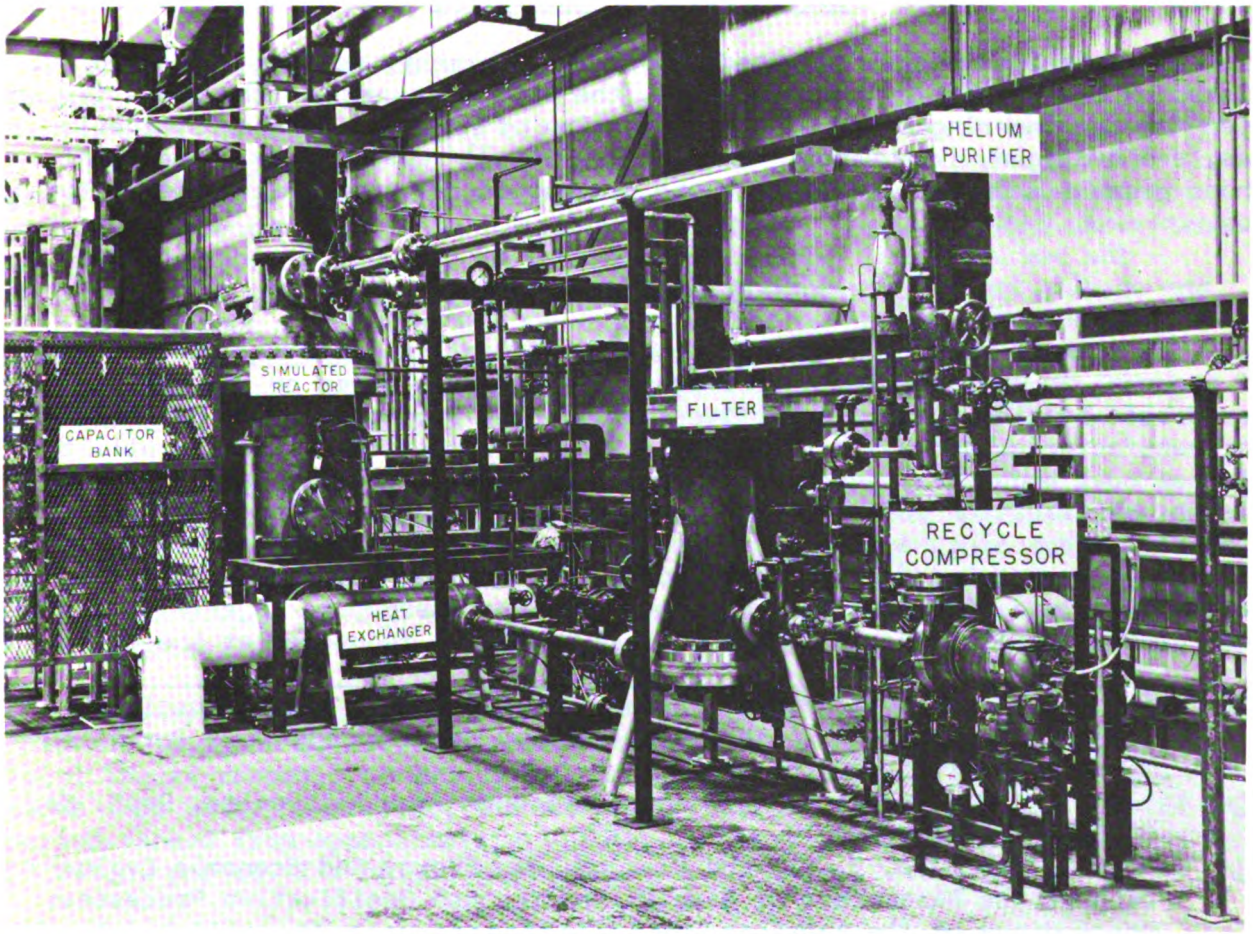

FIGURE 10. - Helium Recycle Loop, 2,500° F., 250 p.s.i.g.

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Construction Materials

To construct nuclear reactors for coal gasification and other industrial purposes, materials must be available that withstand high temperatures and other severe conditions. Many materials have been tested for fuel-element claddings, heat exchangers, and other components. The most promising substances found to date include silicon carbide, molybdenum disilicide, and certain types of graphite, but techniques for the fabrication into large-scale equipment have not yet been developed. Emphasis presently is on testing heat-resistant metals and alloys for heat exchangers.

Purification of Helium

In a high-temperature gas-cooled nuclear reactor, minute traces of gaseous impurities in the recycled helium may react with components of the reactor and cause failure. A tester is being devised to determine trace impurities in helium at concentrations as low as one part per million, and methods are being developed to reduce these impurities to extremely low concentrations (parts per billion).

Gasification of Anthracite

The data from tests conducted at Dorsten, Germany, were analyzed and published by the Bureau.70/ Subsequently, the data were used as the basis for an open-file engineering study on the production of high-B.t.u. gas, synthesis gas, and hydrogen, from Pennsylvania anthracite.71/ This study showed that under present economic conditions, the process is not competitive with natural gas.

Underground Gasification

The primary objective of underground gasification is to recover the energy of the coal as combustible gas for producing electric power or as sensible heat for direct heating. Other objectives include the production of a gas suitable for manufacturing synthetic fluid fuels and other chemicals, and the utilization of coal beds that cannot be mined economically. Several ways of establishing gasification paths in the coal bed (that is, by mining, drilling, hydraulic fracturing, application of high-pressure air, and high-voltage current) have been tried.72/ Following path preparation, the coal is ignited, gas-making fluids (such as air, oxygen, water, or steam) are injected into the underground reaction zone, and the product gases are recovered at the outlet.

70/ Morgan, R. E., Eckerd, J. W., Ratway, J., and Baker, A. F., Lurgi-Gasifier Tests of Pennsylvania Anthracite: Bureau of Mines Rept. of Investigations 5420, 1958, 22 pp.

71/ Kellogg, M. W., Co., Pipeline Gas and Hydrogen From Anthracite, Rept. No. CE-58-189, 1958, 2 vols.

72/ Pears, C. D., Capp, J. P., and Fies, M. H., Energy and Economic Evaluation of the Parameters of Several Underground Gasification Processes: Proceedings of the American Power Conference, vol. 20, published by the Illinois Institute of Technology, 1958, pp. 335-345.

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In the hydraulic fracture tests, significant increase in input and output capacities of the boreholes were noted when small volumes of water were injected. An experiment was therefore conducted to test water injection as the means of preparing the coal seam for linkage. Attempts to link two holes which had been injected with as much as 1,000 gallons of water were unsuccessful owing to the unusually large inflow of water to the outlet borehole and the distance between boreholes, 190 feet apart (about four times the distance between previously linked pairs of boreholes).

To study the action of water injection in the coal bed, a barrier pillar some 500 feet long and 100 feet wide was selected for injection tests under conditions approximating those in the project area, but permitting visual observation of the pillar faces. A water injection borehole was drilled into the pillar at a distance of 50 feet from three sides of the pillar.

More than 2,000 gallons of water were injected into the pillar at a rate of 26 gallons per minute and pressures ranging from 350 to 600 p.s.i.g. The test indicated that the preferred flow of water through the pillar was between the face cleats rather than the butt cleats. Air permeability tests, both before and after water injection, showed that the permeability of the pillar was increased about six times.

In two other experiments, a series of boreholes were installed, equally spaced, on the circumference of a circle with a radius of 50 feet, with one borehole at the center of this circle. In the first of these trials, water and air were injected to increase the permeability of the coal bed. The coal bed was ignited at each of the boreholes on the periphery, and simultaneous path development was begun to the center borehole. In the second experiment, the permeability of the coal bed was such that water injection was not required. The coal bed was ignited in the center borehole, and linkage and path development was toward the boreholes on the periphery. This method of operation permitted greater control of each individual linkage path and resulted in a much more operable system than the first, but showed little difference in amount of coal consumed or quality of gases produced.

Other experiments were conducted with oxygen-enriched air in an attempt to improve the heating value of the product gas. However, considerable operating difficulty was experienced, due to the high temperatures and molten slag which caused interruption of product gas flow at the outlet. Input flows of 50 percent oxygen as high as 1,000 cu. ft. per minute were used and quality of product gases, at times, reached values up to 220 B.t.u. per cu. ft.

Reduced flows and oxygen concentrations prevented entrainment of molten material into the outlet, and permitted continuous operation for nine days. Total inputs ranged from 500 to 850 cu. ft. per minute, and oxygen concentrations from 30 to 50 percent; the quality of the product gases produced in this period ranged from approximately 150 B.t.u. per cu. ft. at the start to about 70 B.t.u. per cu. ft. on the ninth day.

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Gas Purification

Raw synthesis gas produced from coal contains both solid and gaseous impurities, including fly ash, coke, unburned coal, clinker, and refractory materials, as well as carbon dioxide and gaseous sulfur compounds. Before such gas can be processed into fluid fuels or other chemicals the impurities must be removed or reduced.

Removal of Acid Gases

Acid gaseous impurities are removed from synthesis gas by passing raw gas through alkaline absorbents. The effects of eight variables on the rate of absorption of carbon dioxide by ethanolamine in an agitated absorber were determined in a series of factorial experiments[73] to evaluate the economics of this process. Hot potassium carbonate solution was also tested, but with the agitated absorber as presently designed, it was relatively ineffective. The use of hot potassium carbonate solutions developed at the Bureau for separating carbon dioxide and hydrogen sulfide from synthesis gas has been patented.[74]

Removal of Dust

Solids can be removed from gases by means of electrostatic precipitators, but commercial precipitators are not designed for operation at high temperatures and high pressures. Tests conducted on a laboratory precipitator designed for atmospheric pressure show that at 1,500° F. the breakdown voltage is 8,200 volts (only 40 percent of the voltage at 700° F.), and that the operating efficiency decreases; at 500° F. 99 percent of the dust was removed; at 1,500° F. only 95 percent. Manufacturers have stated that pressure affects operations more than temperature. However, development of an electrostatic precipitator for removing solids from gases at 1,500° F. and 5 atmospheres is being attempted.

Dust can be removed also from hot gases by filtration but high pressure losses occur through the filter. Laboratory-scale studies are in progress to determine whether pressure loss can be decreased and the useful life of hot

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73/ Moore, A. S., and Katell, Sidney, New Agitator Absorber for CO_2: Pet. Refiner, vol. 37, No. 3, March 1958, pp. 163-168.

74/ Benson, H. E., and Field, J. H., Australian Patent 206,221 July 11, 1957; Belgian Patents 544,978 Feb. 4, 1956, 555,242 Mar. 15, 1957, 555,241 Mar. 15, 1957; British Patents 725,000 July 22, 1955, 790,287 May 28, 1958, 790,288 May 28, 1958, 790,289 May 28, 1958; Canadian Patent 551,799 Jan. 21, 1958; French Patents 1,076,348 Apr. 21, 1954, 67,641 Mar. 1, 1955, 67,641 Oct. 7, 1957; Indian Patents 53,927 Mar. 28, 1956, 59,685 June 4, 1958, 59,684 June 4, 1958; Italian Patent 567,787 Oct. 16, 1957; Mexican Patent 58,848 May 15, 1958; Pakistan Patents 107,318 Mar. 4, 1958, 107,867 Oct. 6, 1958, 107,901 Oct. 22, 1958; Portuguese 33,820 July 22, 1957; Union of S. Africa Patents 631 Oct. 24, 1955, 537/57 Nov. 20, 1957, 538/57 Nov. 6, 1957; Spanish Patent 234,492 Mar. 30, 1957.

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filters can be extended if accumulated dust is removed periodically by reversing the flow of gas. Tests are being conducted--with three grades of metal filters having a wide range in porosity--at 1,000° F. (the upper limit for the stainless steel used). Short, high-velocity reversed blasts of cleaned gas have proved effective for removing most of the dust accumulated in the filters; however, all the dust is not removed, and increasing pressure loss eventually restricts use of the filters.

The removal of solid impurities from synthesis gases by high-pressure water scrubbing continues to be the most widely used, simplest, and cheapest method available, but little information is available about the effects of variables on the efficiency of such scrubbers. Therefore, this process is also being investigated.

In connection with these studies, a laboratory device is being developed for continuously feeding controlled amounts of finely-divided solids into gases at elevated pressures. Methods are also being developed for sampling gases at elevated pressures and temperatures so that the concentration of solid impurities can be determined. A study has been made of the size of the suspended dust agglomerates as these exist in the gas stream. Tests were made with membrane filters, and the data compiled for publication.

In processing synthesis gas, optimum ratios of hydrogen to carbon monoxide are desired not only to make specific end products but also to permit maximum utilization of the gas. These ratios can be adjusted by treating all or part of the gas in a carbon monoxide shift reactor with chromium-promoted iron oxide catalysts. The activity of these catalytic pellets is reduced when they are covered by dust and attacked by sulfur impurities. The activity can be restored by removing the dust, but sulfur compounds convert the iron oxide to sulfide, and the temperature of the catalyst must be increased slightly to maintain activity. Penetration of dust into the catalyst pellets at 300 p.s.i.g. may cause a permanent reduction in activity; tests at pressures up to 300 p.s.i.g. are in progress to check this point.

Process Economics

Cost studies are prepared for evaluating various phases of the processes of coal utilization being investigated. Several of the studies are concerned with production of synthetic pipeline gas from coal.75/ Two such studies on plants for making synthetic pipeline gas have been completed; in each, synthesis gas is produced in Lurgi fixed-bed gasifiers. One is assumed to operate at 25 atmospheres, the other at 5 atmospheres.

The first step in unit cost evaluation is developing the cost of utilities. Sources of steam for a process plant are discussed and their economics compared in a recent publication.76/ The unit costs of intermediate process

75/ Katell, Sidney, The Economic Outlook for Pipeline Gas from Coal: Coal Age, vol. 63, No. 9, September 1958, pp. 116-118.

76/ Katell, Sidney, and Joyce, T. J., How to Allocate Process Steam Costs: Chem. Eng., vol. 65, No. 5, Mar. 10, 1958, pp. 152-154.

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steps must also be obtained, and the method used has been described in a previous publication.77/ A bibliography on investment and operating costs for chemical and petroleum plants was prepared.78/

COMPLETE CONVERSION OF COAL TO FLUID FUELS

Coal Hydrogenation

Autoclave Studies

Batch autoclaves are used to study reaction mechanisms, catalyst activities, and the effects of primary variables in the hydrogenation of coal, and to provide data for pilot-scale investigations.

In continuing studies of the mechanism of the iron-catalyzed hydrogenation of coal, the hydrogenation of asphaltene was investigated. Asphaltene, a high molecular weight, non-distillable material, is always produced in the reaction between coal and hydrogen, the amount depending on operating conditions. Since the hydrogenation of asphaltene is the slow step in the conversion of coal to a distillable oil, the development of more efficient catalysts could lead to process improvements and also clarify the mechanism of coal hydrogenation. Crude asphaltene was hydrogenated in the presence of iron compounds at 8,000 p.s.i. and 450° C. for 15 minutes. Both ferrous and ferric chloride were active and better than six other iron compounds tested. The effect of hydrogen sulfide is important because appreciable amounts are formed during coal hydrogenation from the sulfur in coal. Added hydrogen sulfide decreased the activity of the chlorides but increased the activity of almost all of the other iron compounds tested.

Model compounds were hydrogenated to determine which reactions are catalyzed by iron and iron compounds in coal hydrogenation. Results indicate that ether linkages and organic sulfur in coal are removed by heterogeneous catalysis over ferrous sulfide. Iron was more active but poisoned by sulfur. Ferrous chloride was inactive. The activity of ferrous sulfide decreased in the presence of hydrogen sulfide and increased in the presence of small amounts of water.

A study of molybdenum-catalyzed coal hydrogenation was made to determine the chemistry involved and also the maximum activity attainable with molybdenum. Experiments with Rock Springs coal indicated the promotional effect observed with sulfuric acid is not related solely to the alkalinity of coal but may also involve interaction of acid and molybdenum. The comparative activities of various molybdenum compounds were also determined.

Hydrogenation of coal in the presence of sodium hydroxide results in high conversions and the production of large amounts of sodium carbonate. On the

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77/ Katell, Sidney, and Joyce, T. J., Determining Process Unit Operating Costs: Cost Eng., vol. 3, No. 4, October 1958, pp. 103-108.

78/ Katell, Sidney, Bibliography of Investment and Operating Costs for Chemical and Petroleum Plants, July 1954-December 1956: Bureau of Mines Inf. Circ. 7847, 1958, 100 pp.

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other hand, experiments with Rock Springs coal showed that sodium carbonate was noncatalytic with or without added water. Unsuccessful attempts were made to separate spent catalysts from residues by froth flotation and float-and-sink techniques. The catalyst is apparently uniformly dispersed throughout and bonded to the organic material. Attempts will be made to separate the catalyst from the coal residue by grinding followed by float-and-sink separation.

Pilot Plant

Low-Pressure Hydrogenation

A study is directed to the development of a low-pressure (2,500 p.s.i) process for converting coal into distillable oils that could be used for gas turbines, jet aircraft, and industrial and domestic heating.

To make gasoline, coal is normally hydrogenated in two steps at 8,000 and 10,000 p.s.i. that require large quantities of hydrogen, power, supporting facilities, and labor. The production of a distillable fuel by a single-stage liquid-phase hydrogenation at low pressure would be more favorable economically. Since distillable oil is almost equal in value to gasoline, attention is being directed to the development of processes for producing these fuels rather than gasoline.

Hydrogenation of coal to heavy fuel oil at 1,500 p.s.i. pressure was previously demonstrated. Conversion to distillable products at 2,500 p.s.i. pressure is much more difficult because the hydrogenation of asphaltic heavy oils is slow at low pressures. Studies were continued in a small, continuous pilot plant at 465° C. and 2000 p.s.i.g. to determine the effect of decreasing concentrations of molybdenum or iron catalysts at lower throughputs. Concentrations as low as 0.1 percent molybdenum permitted adequate hydrogenation of coal and asphaltenes at a throughput of 13 pounds of coal per cubic foot of reaction space per hour. Over 90 percent of the coal was converted to liquid and gases; the yield of oil, including C_4-C_6 hydrocarbons, was 56 percent, and the yield of C_1 to C_3 gaseous hydrocarbons was 18 percent. The liquid had a 90 percent distillation (ASTM) point of 334°C. The use of either 1 or 0.5 percent molybdenum at the same throughput resulted in about the same yields which were slightly greater than the yield obtained with 0.1 percent molybdenum at a lower throughput.

Ferrous sulfate at a concentration corresponding to 2 percent iron on coal, satisfactorily catalyzed the conversion of Rock Springs coal to distillable products. At a throughput of 13 pounds of coal per cubic foot of reaction space per hour, the yield of oil was 62 percent and gaseous hydrocarbons 16 percent, based on m.a.f. coal. The distillation range and the chemical composition of the product were essentially the same as those produced with molybdenum.

Coal could also be hydrogenated to distillable oil with 1 percent iron (added as sulfate) on coal, by decreasing the hourly feed rate to 9.3 pounds of coal per cubic foot of reaction space. The yield of oil was about 60 percent

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and of C_1 to C_3 gaseous hydrocarbons about 20 percent. Thus, the yield with 1 percent iron was greater than with 0.1 molybdenum and almost as large as the yield obtained with 0.5 or 1 percent molybdenum, although the optimum throughput for iron was lower.

Autoclave tests have recently shown that ferrous chloride is much more effective than other iron compounds (and even tin, nickel, or molybdenum), for converting asphaltenes to oil, although it is less active for converting coal to asphaltenes. The results of the autoclave and pilot plant tests suggest the use of both ferrous sulfate and ferrous chloride for the hydrogenation of coal at low pressures in an integrated operation where sulfate would be added to fresh coal for conversion to asphaltenes, and chloride would be added in the reaction zone for conversion of asphaltenes to lower molecular weight products.

High-Temperature Hydrogenation

Experiments were conducted at temperatures up to 800° C. at 6,000 p.s.i.g. in a small, semicontinuous bench-scale unit to determine whether a bituminous C coal (Rock Springs, Wyo.) could be hydrogenated rapidly to appreciable quantities of gaseous hydrocarbons and low-boiling oils. The equipment permitted rapid heating to a given temperature. With 1 percent molybdenum (added as ammonium molybdate) on m.a.f. coal, the yield of oil increased from 2 percent at 450° C. to a maximum of 20 percent at 500° C., decreasing sharply to 4-1/2 percent at 800° C. and remaining constant at this temperature even after 15 minutes. The yield of gaseous hydrocarbons increased from 1 percent at 450° C. to 8 percent at 500° C. and 38 percent at 800° C. After 15 minutes at 800° C. it was 80 percent, corresponding to a yield of about 40,000 cubic feet of methane per ton (m.a.f.) of coal. At 800° C. coal is apparently carbonized very rapidly to char, which is then hydrogenated at a moderate rate to gaseous hydrocarbons without production of additional oil. Catalysts increase the yields of oil and gaseous products between 450° and 700° C. but have no effect between 700° and 800° C.

With 1 percent molybdenum and increased residence time at 480° or 525° C., the yields of gaseous products and oil increased, the rates of formation being most rapid during the first 2-1/2 minutes at temperature.

About 60 percent of the oils produced at 800° C. boiled below 355° C., regardless of residence time. Half of the low-boiling fraction consisted of single-ring aromatics (largely benzene) and a third of naphthalene. At 600° C. and lower, only small quantities of low-boiling oils and little benzene and naphthalene were produced.

Unsuccessful attempts were made to carbonize Rock Springs coal at 600° C. in helium for 4 hours, and to hydrogenate the resultant chars (30 by 60 mesh) at 800° C. and 6,000 p.s.i.g., with and without catalysts. The temperature within the char bed increased to above 1,000° C. after less than 1 minute at 800° C., and the reactor ruptured. Experiments are planned to develop methods of controlling char hydrogenation and to determine the effect of time, pressure, and temperature on the production of gaseous hydrocarbons.

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To evaluate the hydrogenation of char and coal to gaseous products more completely, a small continuous unit was designed to operate at 800° C. and 6,000 p.s.i., with coal fed at pressure from a hopper into a stream of hydrogen and transported into a tubular reactor. Retention time will be 2 to 20 seconds, and hourly feed rates will be 50 to 100 grams of coal in the initial experiments. The apparatus has been completed, and the coal feeder is now being developed.

A high-volatile bituminous C coal (Rock Springs, Wyo.) was heated to 800°-1,500° C. for several milliseconds by a high-intensity flash lamp to determine degradation products in hydrogen, in vacuo, and with iodine in vacuo. The products appeared to be gaseous, and little, if any, oil was produced. Quantitative yields could not be determined; water, hydrogen, carbon monoxide, acetylene, methylacetylene, and traces of hydrogen cyanide and carbon disulfide were detected. The latter four compounds are uncommon to conventional hydrogenation or carbonization processes.

A patent was granted for a process involving the liquid-phase hydrogenation of coal at 500°-525° C.[79]

Characterization of Hydrogenation Products

The usefulness of the mass spectrometric technique for analyzing liquids was increased by combining low-ionizing-voltage and type-analysis methods. A low-ionizing voltage produces a mass spectrum consisting of only molecular mass peaks. In type-analysis, fragmentation peaks, characteristic of different classes of compounds, permit quantitative determination of types. By applying both methods, one can determine the types of compounds present, and the total percentage and molecular weights of the homologs of each structural type.

The low-voltage method has been applied principally to tar acid fractions of oils derived from hydrogenation and carbonization of coal,[80] and to coal-tar distillates subjected to dealkylation. A calibration program is now in progress to apply the low-voltage method to quantitative determination of phenolic types.

A rhenium filament as an electron emitter in the ion sources of mass spectrometers has been developed by the Bureau and has improved the stability and efficiency of the instrument for analysis of high-molecular weight materials.[81] The instability encountered with carbided tungsten filaments has been eliminated. Because spectra are more reproducible and filaments last longer, rhenium filaments are now used in most analytical mass spectrometers in this county.

79/ Pelipetz, M. G., Balanced Hydrogenation of Coal: U.S. Patent 2,860,101, Nov. 11, 1958.

80/ Sharkey, A. G., Jr., Wood, Gorton, and Friedel, R. A., Mass Spectrometric Analysis of High-Boiling Coal-Hydrogenation Products Using Low-Ionizing Voltage: Chem. and Ind., No. 26, June 28, 1958, pp. 833-834.

81/ Robinson, C. F., and Sharkey, A. G., Jr., Rhenium as an Electron Emitter in Mass Spectrometry: Rev. Sci. Instr., vol. 29, March 1958, pp. 250-251.

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Gas chromatography was also applied to products from coal hydrogenation. Mixtures of m- and p-xylene were separated by means of columns wetted with tetrahalophthalates.[82] M- and p-cresol were separated chromatographically for the first time by converting the phenols to their trimethylsilyl ethers[83] and passing these over either silicone or di-n-butyl tetrachlorophthalate columns.[84]

Fischer-Tropsch and Related Syntheses

An alternative method to coal hydrogenation for production of synthetic liquid fuels involves intermediate gasification of coal to synthesis gas containing carbon monoxide and hydrogen, with subsequent conversion to liquid products by catalytic reactions.

Chemistry of Metal Carbonyls

Investigations in carbonyl chemistry have elucidated the structure of a number of compounds similar to intermediates that have been postulated in the Fischer-Tropsch synthesis. The manner in which new carbon-carbon bonds are formed in reactions catalyzed by iron and cobalt complexes is being investigated.

Carbonyl groups in phthalic and acetic anhydrides and in diphenylurea have been found to exchange with gaseous carbon monoxide when heated with catalytic amounts of dicobalt octacarbonyl, $Co_2(CO)_8$, at 100° to 200° C. The compounds that exchange with carbon monoxide also undergo hydrogenolysis when treated with synthesis gas ($1H_2+1CO$) in the presence of $Co_2(CO)_8$. The exchange reactions probably proceed through the formation of intermediates containing carbon-metal or nitrogen-metal bonds, followed by insertion of carbon monoxide between the atoms forming this bond.[85]

The simple concept that reactions of CO catalyzed by transition metals involve the insertion of CO between the atoms of a metal-carbon or metal-nitrogen bond has furnished an explanation of the course of most carbonylation

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82/ Langer, S. H., Zahn, Charles, and Pantazoplos, Gus, Gas-Liquid Chromatographic Resolution of m- and p-xylene. Tetrahalophthalate Liquid Phases: Chem. and Ind., No. 35, Aug. 30, 1958, pp. 1145-1147.

83/ Langer, Stanley H., Connell, Samuel, and Wender, Irving, Preparation and Properties of Trimethlsilyl Ethers and Related Compounds: Jour. Org. Chem., vol. 23, No. 1, Jan. 1958, pp. 50-58.

84/ Langer, S. H., Pantages, Peter, and Wender, Irving, Gas-Liquid Chromatographic Separation of Phenols as Trimethylsilyl Ethers: Chem. and Ind., No. 50, Dec. 13, 1958, pp. 1664-1665.

85/ Wender, I., Friedman, S., Steiner, W., and Anderson, R. B., Exchange of Carbonyl Groups in Anhydrides with Carbon Monoxide. Mechanism of Carbon Monoxide Transfer to Organic Molecules via Transition Metals: Chem. and Ind., No. 51, Dec. 20, 1958, pp. 1694-1695.

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reactions.86/ This concept also leads to the suggestion of an alternate mechanism for the production of straight-chain molecules in the Fischer-Tropsch synthesis. In the scheme that was developed, steric difficulties may be avoided if the surface is sparsely occupied; also, insertion of CO from a neighboring metal atom may occur.

An organometallic complex with two carbon-metal bonds should add two molecules of CO.87/ Thus, when $Co_2(CO)_6HC{\equiv}CH$ is heated with CO, two new carbon-carbon bonds are formed, one CO adding to each carbon atom of the acetylene molecule. Unsaturated molecules (olefins, acetylenes) are generally thought to be adsorbed on two sites on a metal surface. However, a trinuclear complex, containing one acetylene associated with three metal atoms, has been isolated and its structure elucidated.88/ Thus, more than two atoms on a metal surface may participate in adsorbing unsaturated molecules, and this type of interaction may influence the catalytic behavior of the molecule or lead to carbide formation.

Catalyst Studies

A Fischer-Tropsch catalyst that could be used at temperatures of 300° to 450° C. would give improved product distribution, greater productivity, permit the use of catalysts of low specific activity, and yield more valuable byproduct steam. However, carbon deposition has hindered the development of such a catalyst. At higher temperatures, this reaction plugs reactors and causes spalling of the catalyst. Hence this temperature range has been avoided by many investigators.

These temperature conditions are being investigated with a highly reactive residual catalyst that has been tentatively identified as material produced by the action of carbon monoxide or synthesis gas on iron. This material appears to have an activity that corresponds to the activity obtained when 50 to 100 times as much of a conventional catalyst is used. Because of the remarkable catalytic activity of this carbon-expanded iron, efforts are being made to develop it into a practical catalyst. Synthesis tests with three types of carbon-expanded iron have been successful:

1. Carbonaceous material from a bed of conventional iron catalyst that was blocked by carbon deposition at high temperatures was dispersed on Pyrex glass wool.

86/ Greenfield, Harold, Metlin, Sol, Orchin, Milton, and Wender, Irving, Hydrogenation of Thiophene Compounds with Hydrogen and Carbon Monoxide and a Cobalt Catalyst: Jour. Org. Chem., vol. 23, No. 7, July 1958, pp. 1054-1056.

87/ Sternberg, H. W., Markby, R., and Wender, I., A Quinone Iron Tricarbonyl Complex and Its Significance in Organic Synthesis: Jour. Am. Chem. Soc., vol. 80, No. 4, Feb. 20, 1958, pp. 1009-1010.

88/ Markby, Raymond, Wender, Irving, Friedel, Raymond A., Cotton, F. A., and Sternberg, Henry W., The Association of an Acetylene with Three Metal Atoms; A New Trinuclear Cobalt Complex: Jour. Am. Chem. Soc., vol. 80, No. 24, Dec. 20, 1958, pp. 6529-6533.

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2. Finely divided electrolytic iron was dispersed on Pyrex glass wool and activated with synthesis gas in a stainless steel reactor at approximately 400° C., presumably by forming a carbon deposit.

3. A carbon deposit was developed on the inside of an otherwise empty carbon-steel reactor by treatment with synthesis gas at 300 p.s.i.g. and about 400° C.

All of these catalysts were active for many weeks in synthesis at 300 p.s.i.g., hourly space velocity of 300, and 325° C. Products from a fused iron catalyst and from carbon-expanded iron were compared, and the latter yielded a lower molecular weight product containing essentially no wax. The olefin content of both liquid and gaseous hydrocarbons was high, and the liquid fractions contained only traces of oxygenated molecules.

Sulfur Poisoning

The poisoning effect of sulfur compounds on Fischer-Tropsch catalysts is being studied to determine an economic balance between gas purification and catalyst replacement, and for a better understanding of the nature of poisoning.

Two techniques are being employed, liquid-phase poisoning, that is, pretreating the catalyst in a solution of a sulfur compound; and gas-phase poisoning—adding sulfur compounds to synthesis gas. Hydrogen sulfide, sulfur dioxide, carbon oxysulfide, carbon disulfide, and ethyl mercaptan have been used.

Gas-phase poisoning is more effective in decreasing catalytic activity because it affects only the outer parts of the catalyst particles that are active in the synthesis, whereas liquid-phase poisoning occurs more uniformly within the particle. Only 0.2 mg. or less of poison in the gas phase reduced the activity 50 percent, while 1 to 4 mg. of the same poison was required in the liquid phase to effect the same decrease in activity.

Products with poisoned Fischer-Tropsch catalysts have been studied by gas chromatography, and the results indicate that sulfur reacts preferentially with alkali on the catalyst surface. Adsorption studies have confirmed the reaction of sulfur compounds with the alkali promoter.

The investigation of sulfur poisoning of Fischer-Tropsch catalysts required new methods for storing synthesis gas containing known concentrations of sulfur compounds. Conventional steel cylinders reacted with many sulfur compounds. Stainless steel gas cylinders were satisfactory but expensive. Cylinders coated with a phenolic resin reacted slowly to produce small changes in concentration that could be tolerated in most experiments.

Other Catalyst Studies

Kinetic data for the Fischer-Tropsch synthesis on nitrided synthetic ammonia catalysts at 21.4 atmospheres with gases ranging from $2H_2+1CO$ to

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$0.25H_2+1CO$ were evaluated.[89] These catalysts maintain essentially constant activity over long periods. The overall activation energy increased with the carbon monoxide content of the feed gas from 19.5 kcal/mole (for $2H_2+1CO$) to 23.7 (for $0.7H_2+1CO$).

At the request of Ullmann's Encyklopädie der technischen Chemie (publishers Urban and Schwarzenberg, München--Berlin, Germany) two manuscripts were written, summarizing Fischer-Tropsch data obtained by the Bureau of Mines, for inclusion under "Carbon Oxide Hydrogenation" in volume 9, pp. 684-748. One of these papers was published in a German journal.[90]

A translation of Russian investigations of the mechanism of the Fischer-Tropsch synthesis was published.[91] Three book reviews were published.[92 93 94]

Pilot Plant

Because the Fischer-Tropsch synthesis releases large amounts of heat, development of suitable reactors is a major engineering problem. The status of the art was reviewed.[95]

A hot gas recycle process, designed so that large volumes of product gas are circulated through the bed of catalyst to remove the heat of reaction, is being studied on a pilot-plant scale. Reactor design has been simplified, as no heat exchange surface is required in the catalyst bed. The sensible heat of the recycle gas is recovered by generating high-pressure steam in an external conventional waste-heat boiler.

89/ Karn, F. S., Seligman, B., Shultz, J. F., and Anderson, R. B., Kinetics of the Fischer-Tropsch Synthesis on Iron Catalysts. I. Rate Data on Reduced and Nitrided Catalysts: Jour. Phys. Chem., vol. 62, No. 9, Sept. 1958, pp. 1039-1043.

90/ Anderson, R. B., Hofer, L. J. E., and Storch, H. H., Der Reaktionsmechanismus der Fischer-Tropsch-Synthese (Reaction Mechanism of the Fischer-Tropsch Synthesis) Chemie-Ingenieur-Technik, vol. 30, No. 9, Sept. 1958, pp. 560-566.

91/ Eidus, Y. T., Mechanism of the Synthesis of Hydrocarbons From Carbon Monoxide and Hydrogen: Bureau of Mines Inf. Circ. 7821, 1958, 13 pp.

92/ Anderson, R. B., Book Review, Chemisorption: Proceedings of Symposium, College of North Staffordshire, Keele, Staffordshire: Chem. Soc., July 16-19, 1956; Acad. Press Inc., New York, N. Y., 1957; Arch. Biochem. and Biophys., vol. 77, No. 1, Sept. 1958, pp. 243-244.

93/ Hofer, L. J. E., Book Review (Advances in Catalysis and Related Subjects Vol. IX): Jour. Am. Chem. Soc., vol. 80, No. 10, May 20, 1958, pp. 2598-2599.

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Previous operation of the pilot plant had demonstrated that heat could be effectively removed and catalyst temperature controlled up to about 325° C. At an hourly space velocity of 1,000, a recycle ratio of 45, and a feed-gas conversion of 90 percent, the temperature increase in the bed was 20° C., and the pressure drop was 1 to 1.5 p.s.i. per foot of catalyst bed. During the past year, attention was directed to decreasing the cost of recycling by using catalysts that offered lower resistance to gas flow or by decreasing the recycle ratio, and improving product quality.

Commercial steel shavings and lathe turnings were used as catalysts, packed with void volumes of 96-97 percent, thus offering very low resistance to flow. The catalysts were partly oxidized at 600° C. with steam, impregnated with potassium carbonate, and reduced with hydrogen at 400° C. Although the pressure drop through the bed was small, activity was too low. Lathe turnings of 88-percent voids (and approximately 3 times the steel content of the 96-percent void turnings) were used in subsequent studies of activation in synthesis. No special gas composition was needed for catalyst activation--good activity was obtained with $1.4H_2+1CO$. When the ratio of recycle to fresh gas was lowered from 45 to 17, the temperature gradient increased from 20° to 40° C., and the pressure drop through the bed decreased to less than one-third of its previous value. This decrease in pressure drop would substantially reduce the equipment and compression costs.

An attempt was also made to upgrade the hydrocarbon products, passing about 3 percent of the recycle gas stream through a reformer containing activated alumina. Product distribution was not changed significantly.

To determine the applicability of the hot-gas-recycle system to the synthesis of methane on steel turnings, short tests were made with feed gas having H_2:CO ratios of 2 and 3. Over 90 percent conversion of the hydrogen and carbon monoxide was achieved at an hourly space velocity of 1,000, a gas recycle ratio of 44, and 320° C.—90 percent of the product was gaseous hydrocarbons.

The gross heating value of the noncondensable gases was 850 B.t.u. per cu. ft. Excellent temperature control was achieved during these tests, so that the hot-gas-recycle system should be suitable for methanation. The heating value of this gas could be significantly improved by reacting residual hydrogen with CO and CO_2 to produce additional methane in a subsequent pass through a bed of nickel catalyst.

Characterization of Synthesis Products

Mass spectrometry has been applied to the gaseous, oil, and wax fractions of Fischer-Tropsch products. Trimethylsilyl derivatives are used for alcohol determinations by mass spectrometry.[96] A method is being developed for sepa-

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rating normal and branched saturated hydrocarbons in high-boiling oils by molecular sieves at elevated temperatures.

Gas chromatography[97 98] should permit comprehensive analysis of coal-to-oil products. Techniques for the application of this tool to the analysis of Fischer-Tropsch hydrocarbons are being developed; a detailed analysis of hydrocarbons up to C_{10} is in progress. The reaction product has been separated by distillation and liquid chromatography, and analysis of the C_6 fraction by a combination of gas chromatography and mass spectrometry is almost complete.

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